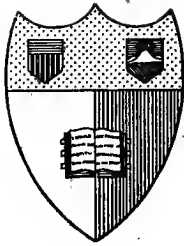


LUBRICATING OILS,  
FATS AND GREASES

GEORGE H HURST

PUBLISHED AT THE OFFICES OF THE  
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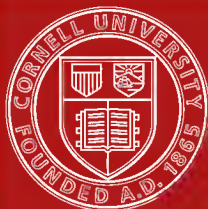
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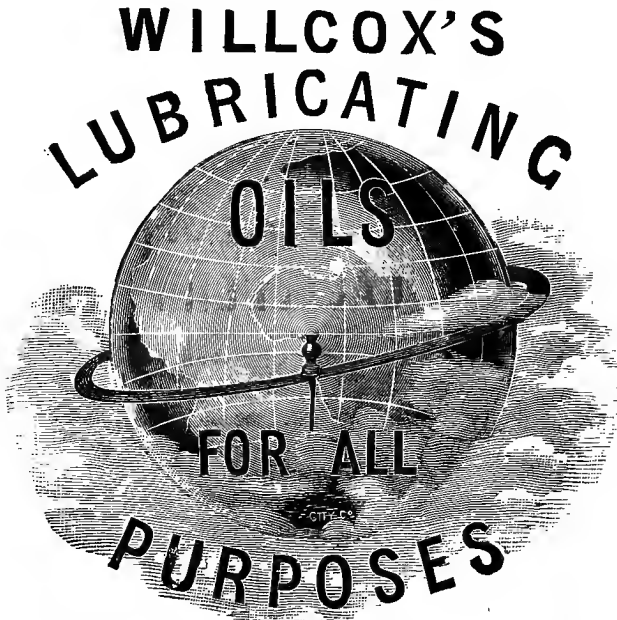
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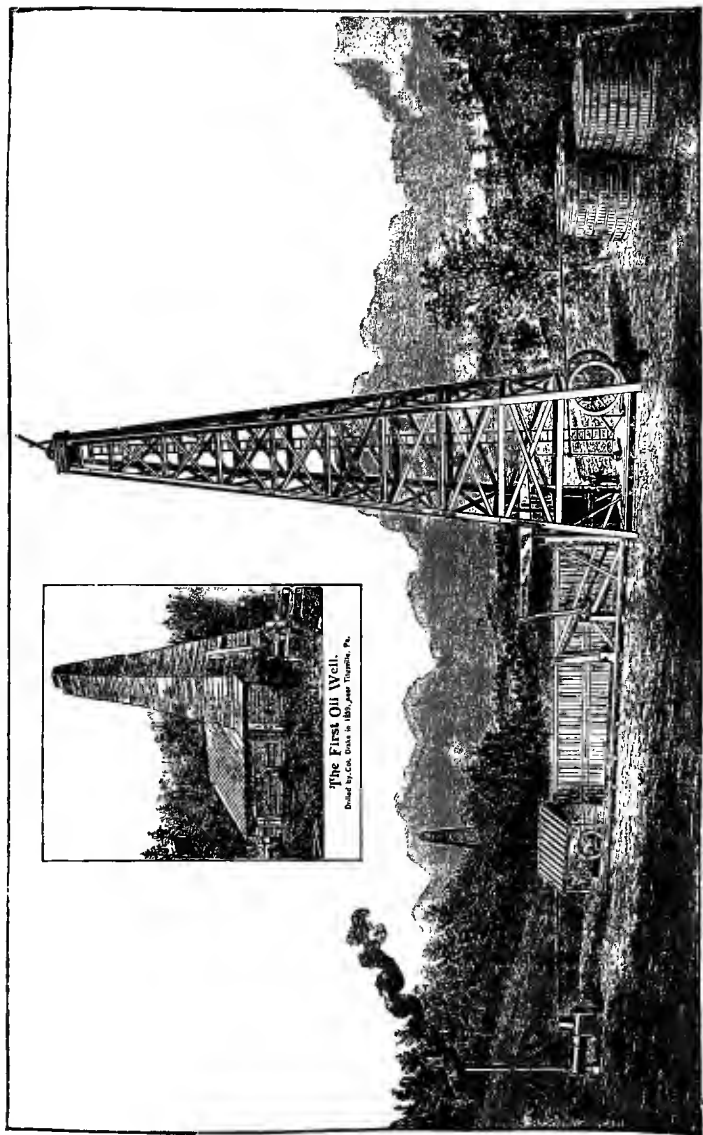
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## LUBRICATING OILS, FATS AND GREASES

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Relative dimensions of the machinery of the first oil-well, and one of recent date. The derrick of the former, bored in 1859, was 34 feet high, the height of the modern derrick (1891) being 82 feet. [To face p. 76.]





# LUBRICATING OILS, FATS

AND

## GREASES.

THEIR ORIGIN, PREPARATION, PROPERTIES, USES,  
AND ANALYSIS.

BY

GEORGE H. HURST, F.C.S.

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LONDON:  
SCOTT, GREENWOOD & CO.

19 LUDGATE HILL

*Publishers of the Oil and Colourman's Journal.*

1896.

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## PREFACE.

THIS little book has been written with the object of supplying oil dealers and users with some information as to the various oils which are used for the purpose of lubricating machinery, and this object has been kept in view throughout, and the special properties of various products which cause them to be of value as lubricants are particularly pointed out; while only those oils which are at all in extensive use for this purpose are mentioned, the mineral or hydrocarbon oils, which have largely, if not entirely, displaced the fatty oils, having special attention given to them.

It is not intended that this book should be a full treatise on the preparation of the various oils, but some information which it is thought will be sufficient for the purpose is given on this subject.

The chapter on the Analysis of Oils is not written with a view of making the reader an expert oil analyst, but is confined to giving some details of the best methods of testing oils with a view of enabling the dealer or consumer to make ordinary and regular tests of the quality of his oils, while he is recommended to engage the services of an analyst in special cases which may arise.

In the chapter on Lubrication an endeavour has been made to deal with the principles of that

important subject ; unfortunately, information is lacking as to the real value of various oils in the lubrication of machinery, and any one who is in a position to do so would confer a favour on machinery users by making a series of observations on this subject and giving his results to the world.

GEORGE H. HURST.

CHEMICAL LABORATORY,  
22 BLACKFRIARS STREET, SALFORD,  
*October, 1896.*



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# LUBRICATING OILS.

## CHAPTER I.

### INTRODUCTORY.

#### OILS AND FATS.

OILS and fats are a group of valuable bodies employed for a great variety of purposes, formerly obtained solely from animal or vegetable sources, but of late also from mineral sources, which have certain special characteristics that distinguish them from other bodies. An oil is a liquid body, a fat is a soft solid body; but often it is purely a question of temperature or climatic conditions as to whether a particular product be an oil or a fat. Olive oil is in the country of origin always a liquid, but in this country we are familiar with the fact that in the winter-time it sets into a solid fat, while in Iceland it would be considered a fat pure and simple; coconut oil in Ceylon, where it is largely extracted, is a water-white fluid oil; here it is always a solid fat. Other examples could be quoted to show that the distinction between oils and fats is but a nominal one.

The special features which distinguish the oils and fats from all other groups of compounds are:—

1. They are mostly liquid bodies which are lighter than water, the specific gravity ranging from 0.730 to 0.980. Fats are also lighter than water.

2. They are viscous bodies as a rule. Some of the lighter oils or products which will be dealt with are limpid like water, but such are only grouped with the oils on account of their other general features ; viscosity is the characteristic property of an oil.

3. They impart a transparent greasy stain to paper, which is permanent as a rule.

4. They are quite insoluble in water, but slightly so in alcohol, although in this respect they vary very much ; castor oil is completely soluble in alcohol, olive oil but partially so ; the hydrocarbon oils are insoluble. The oils and fats are readily soluble in ether, carbon bisulphide, turpentine, benzol, chloroform, and some other solvents of a similar character ; with the single exception of castor oil, they are all soluble in petroleum spirit.

Oils are divisible into two large groups :—

(1) Fatty oils and fats.

(2) Hydrocarbon oils.

1. *Fatty Oils and Fats*.—It will be sufficient in this place to mention that these oils are compounds of the three elements, carbon, hydrogen and oxygen ; while this, however, is the ultimate chemical composition of the oils and fats belonging to this group, they may be resolved into simpler bodies than themselves by a process of proximate analysis, *viz.*, into glycerine, and one or more bodies of an acid nature, which are hence known as fatty acids. In another chapter this question will be dealt with in detail. All the fatty oils and fats are derived from the animal and vegetable kingdoms of nature ; while some are liquid and are then known as oils, others are solids, generally of a soft consistency like butter, and then they are called fats ; but, as pointed out above, the distinction between an oil and a fat is usually brought about by climatic conditions.

When boiled with a solution of either caustic soda or

caustic potash, oils and fats undergo a chemical change, resulting in the production of what is known as soap together with glycerine. This change is termed saponification; at one time the term was restricted to that change brought about by the action of alkalis, but it has since been extended by chemists to include the decomposition of fats, etc., into glycerine and fatty acids, no matter by what means this change is brought about. Glycerine is obtained only from fats and oils, hence these bodies are often termed glycerides to indicate this fact.

2. *Hydrocarbon Oils*.—What were originally and often now are so called mineral oils, but which are better classed under the term hydrocarbon oils, are derived wholly from the shale and crude petroleums of the mineral kingdom. They contain only the two elements, carbon and hydrogen. These two elements have the singular property of combining together in varying proportions to form a large number of compounds, known as “hydrocarbons,” of which more will be said presently. The hydrocarbon oils, which will be considered here, have widely varying properties; some are very volatile bodies, water-white in colour and very limpid; others are pale yellow but fluid; while others again are of a somewhat deeper colour, and rather viscid in character; while others again are of a buttery consistence at the ordinary temperature. Some of the hydrocarbons, which will be considered, are of a wax-like nature, and form the valuable product, paraffin wax. The lighter of these products are very inflammable, and will burn freely at the ordinary temperature; such are chiefly employed as solvents in paint and varnish making; others do not burn quite so readily, but when burnt with a wick in a lamp give a good white light, and hence find considerable employment as burning or illuminating oils. The heavier oils are employed in the lubrication of machinery, for which purpose they have largely supplanted

the vegetable and animal oils. One feature of these hydrocarbon oils, which distinguishes them from the fatty oils, is that they are not acted upon by caustic soda or caustic potash. They also possess some amount of fluorescence or bloom, varying according to the source from whence the oil was derived; such fluorescence is not possessed by any fatty oils.

Besides the fatty oils, vegetables and plants often contain other bodies of an oily nature, which possess a characteristic taste or odour closely resembling the characteristic odour or taste of the plant from whence it came; and it is obvious that to these oily products the plant owes its characteristic odour or taste. These have been named the "essential oils," and they differ markedly in their properties and composition from the fatty oils. Sometimes a plant will yield both kinds of oil—a familiar example being the mustard. From this can be expressed by pressure a smooth bland oil possessing all the characters of a fatty oil. Then by distillation there can be obtained a white or almost white oil, which possesses in a marked degree the peculiar odour and taste of the mustard. This is the essential oil of mustard. These oils will not be dealt with in this book.

Oils are used for a great variety of purposes: in lubricating machinery, illuminating, soap-making, food, medicine, etc., etc. In each particular use, certain properties and certain oils are brought into play. For lubricating machinery dependence is placed on their viscid nature and their smooth qualities; in illuminating, the fact that they are combustible; for soap-making only the fatty oils are available, because they are the only ones which can be saponified. Certain oils are employed for food on account of their pleasant taste or odour; while in medicine oils and fats are convenient vehicles for applying many remedies to the human body, and

in certain cases, as in burns or scalds, they have a soothing tendency which makes them act beneficially. A book which shall deal with oils and fats in all their applications has far too wide a scope. Hence the present work will be restricted almost entirely to considering oils and fats from the point of view of their use as lubricants.

## CHAPTER II.

### HYDROCARBON OILS.

THE *hydrocarbon oils*, or, as they are frequently termed, mineral oils, are obtained from two sources:—

1. From oil shales found in Scotland and elsewhere.
2. From petroleum found in America, Russia and elsewhere.

It is owing to the fact that they were first of all obtained from the Scotch shale that they obtained their name of "mineral oils," and which is still used to designate them without distinction as to their origin. Their name of hydrocarbon oils comes from the fact of their being mixtures of various compounds of carbon and hydrogen.

The hydrocarbon oils are produced by a process of distillation from the raw material, of whatever origin that might be; and, as the products obtained are different from the materials employed, the distillation is essentially a destructive one resulting in the production of new compounds.

*Distillation.*—Many bodies are capable of existing in more than one of the forms in which bodies make their appearance in nature. Water, for instance, is known to be a solid, a liquid, or a gas—according to the circumstances under which it exists at the moment. Alcohol can exist as a liquid or as a gas. Benzol, turpentine, aniline, ether, chloroform, are also examples of bodies which are known in two forms. Generally, indeed, it is always simply a question of tempera-



ture which determines whether a body shall be a solid, or a liquid, or a gas. Sometimes pressure influences the conditions in a manner which will be indicated presently.

When a solid body is heated, it begins generally to liquefy, the temperature at which it becomes liquid being called its melting point. This varies considerably with different bodies. Some melt below  $0^{\circ}$  C., others but little above, while others have a high melting point, as, for instance, copper, iron and most metals. Other bodies again cannot be melted with the means at present at the disposition of the chemist; such are said to be infusible.

Liquids, when heated, enter into ebullition, and pass off into the state of vapour or gas. The temperature at which they do this is called the boiling point. This point varies very much. Some liquids boil below the freezing point of water, others at temperatures but little above, while some boil at temperatures above that of water. Some bodies which are solid at the ordinary temperature liquefy on heating, but do not give off any gas at any higher temperature. There are some solid bodies, *e.g.*, ammonium chloride, which pass at once from a state of solid into that of gas on heating.

If the vapour of a liquid be passed through an apparatus, by means of which it can be cooled, it will condense back again into the liquid form in which it originally existed. This is called distillation. A distilling apparatus consists essentially of three portions: first, a retort, or still, in which the body may be heated to convert it into vapour or gas; the second portion consists of an arrangement for cooling the vapours—this is called the “condenser”; while the third and last portion consists of a vessel to collect the condensed liquid or distillate as it is called in.

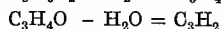
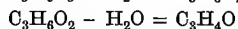
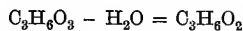
Distillation is of two kinds, simple and destructive. When the distillate possesses all the characteristics of the

original body, in fact, when all that the heat applied has done is to convert the body into vapour which is condensed again in the other portions of the plant, we have a case of simple distillation. On the other hand, when the products of distillation are different from, and have evidently been produced by, the destruction of the original substance, then it is a case of destructive distillation.

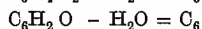
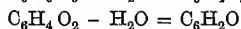
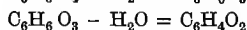
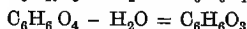
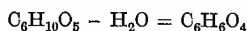
Simple distillation always occurs with bodies of simple molecular composition, as, for instance, with water, alcohol, benzol, turpentine, etc. On the other hand, such bodies as the oils and fats, starch, wood, coal, etc., whose molecular composition must be of a most complex character, cannot be distilled without undergoing destructive distillation, without their molecules being broken up into two or more compounds of a simpler composition.

It has been pointed out that, in all cases of destructive distillation, there is a tendency for one of the elements in the body to remain behind in the retort, or still, in greater proportion than what it exists in the original compound. Mills has called it a case of "cumulative resolution".

Take, for instance, glycerine or glycerol. This on being distilled loses water, and passes into what is called a poly-glycerine, the final product being a hydrocarbon according to the equations:—



In the case of cellulose, we may suppose that action goes on in accordance with the following equations:—



The ultimate result being carbon, which is also the ultimate result obtained in distilling coal, shale, peat, wood and other carbonaceous materials.

In actual practice the products obtained in the destructive distillation of wood, etc., are not so simple as are represented here; while much water is always given off, some of the carbon comes over in combination with the hydrogen as hydrocarbon compounds; some comes over in combination with both hydrogen and oxygen in the form of acid or phenolic compounds, such as acetic acid, phenol, cresylic acid, etc.

The nature of the products obtained in the destructive distillation of bodies like coal, wood, etc., depends upon, first, the composition of the body, and secondly, the temperature at which the distillation takes place.

First, as to the composition of the body, wood will naturally give rise to a very different series of products than coal, and the latter different again to shale. The purity of the compound also has some influence; when pure it does not undergo destruction to the same extent as if impure. When an organic compound is mixed with an infusible inorganic compound, like limestone or clay, then a higher temperature is required to distil the body, and a greater decomposition of the body results, and products of a simpler chemical composition are obtained. This is seen in the cases of the shale and coal industries. In the former case the substance dealt with contains much mineral matter of an infusible character, the result being that a wide variety of products are obtained, most of which have a simple chemical composition and constitution. On the other hand, coal is fairly free from any mineral impurity, and the products from it are more complex in character.

Whenever chlorine, sulphur, oxygen and nitrogen are present in carbonaceous bodies, these always tend to cause

the production of compounds containing them ; thus sulphur always causes the formation of sulphides and other sulphur compounds in the distillate, chlorine forms chlorides, oxygen gives rise to the presence of oxy acids and phenols in the products, while nitrogen results in the production of various nitrogen bases. Thus it is that coal, shale or wood, when distilled, yields aniline, pyridine bases, phenol, cresol, sulphides, etc. Often these are a source of trouble to the refiner of the products. More especially is this the case in the shale industry, where the oxygen, nitrogen and other products are more or less of the nature of impurities, and have to be got rid of ; a work which entails some labour and expense to the shale oil refiner. Sulphur in shale and petroleum has also been found to cause a smaller yield of solid paraffin wax being obtained, a feature which is rather undesirable.

The temperature at which the distillation is conducted has a material influence on the character and quantity of the products. When the temperature is comparatively high, as it is in coal gas-making, then there is produced a large quantity of gas of simple chemical composition, containing much hydrogen, methane, ethene, acetylene, and other gaseous bodies. The characteristic products are a series of what are called aromatic hydrocarbons, of which benzene, toluene, naphthalene, anthracene are the most important members ; besides these there are certain phenolic and amido compounds derived from them.

When the operation is carried on at a dull red heat, such as was attained in the early days of the shale oil industry, while a large quantity of gas is produced, there are no aromatic hydrocarbons, but a large quantity of paraffin and olefin hydrocarbons with some ethers, such as pyrene, chrysene, etc., of a rather complex composition, together with certain pyridine bases and phenolic compounds.

At a low red heat, such as is attained in the most modern shale retorts, the products are a small quantity of gas of high illuminating power, a large yield of liquid hydrocarbons of the paraffin and olefin series, together with phenolic compounds and pyridine bases.

The following table shows the characteristic products obtained in the distillation of coal, shale, wood, and petroleum, the most important being shown in *italic* type:—

## PRODUCTS OF DISTILLATION.

<i>Products.</i>	<i>Coal.</i>	<i>Shale.</i>	<i>Wood.</i>	<i>Petroleum.</i>
<i>Hydrogen</i>	<i>large</i>	<i>traces</i>	<i>large</i>	<i>present</i>
<i>Gaseous Hydrocarbons</i>				
Methane C H <sub>4</sub> and Paraffins	<i>large</i>	<i>large</i>	<i>large</i>	<i>present</i>
Olefins	<i>large</i>	<i>large</i>	<i>considerable</i>	<i>present</i>
Acetylenes	<i>present</i>	<i>none</i>	<i>none</i>	<i>none</i>
<i>Liquid and Solid Hydrocarbons</i>				
Liquid Paraffins	<i>small</i>	<i>large</i>	<i>absent</i>	<i>very large</i>
Solid "	<i>traces</i>	<i>considerable</i>	<i>present</i>	<i>moderate</i>
Liquid Olefins	<i>small</i>	<i>very large</i>	<i>none</i>	<i>considerable</i>
" Pseudo Olefins	<i>none</i>	<i>none</i>	<i>none</i>	<i>present</i>
" Acetylenes	<i>present</i>	<i>present</i>	<i>none</i>	<i>none</i>
Benzene, etc.	<i>large</i>	<i>trace</i>	<i>moderate</i>	<i>present</i>
Naphthalene	<i>large</i>	<i>none</i>	<i>moderate</i>	<i>none</i>
Anthracene	<i>moderate</i>	<i>none</i>	<i>none</i>	<i>present</i>
Chrysene	<i>moderate</i>	<i>considerable</i>	<i>present</i>	<i>present</i>
<i>Oxygenated Bodies</i>				
Acetic Acid	<i>present</i>	<i>present</i>	<i>large</i>	<i>none</i>
Methyl Alcohol	<i>none</i>	<i>none</i>	<i>considerable</i>	<i>none</i>
Phenols	<i>large</i>	<i>considerable</i>	<i>moderate</i>	<i>none</i>
Oxyphenols	<i>none</i>	<i>large</i>	<i>large</i>	<i>none</i>
<i>Nitrogenised Bodies</i>				
Ammonia N H <sub>3</sub>	<i>considerable</i>	<i>considerable</i>	<i>none</i>	<i>none</i>
Anilines	<i>present</i>	<i>none</i>	<i>none</i>	<i>none</i>
Pyridines	<i>considerable</i>	<i>considerable</i>	<i>none</i>	<i>none</i>
Acridine	<i>present</i>	<i>none</i>	<i>none</i>	<i>none</i>
Carbazol	<i>present</i>	<i>none</i>	<i>none</i>	<i>none</i>
Sulphur Compounds	<i>present</i>	<i>present</i>	<i>none</i>	<i>present</i>

The characteristic products are shown in *italic* type.

## HYDROCARBONS.

There are a very large number of compounds of carbon and hydrogen. Their study is much simplified by the fact

that they can be divided into groups or families, in which there is a certain definite relationship between the proportions of carbon and hydrogen contained in members of each group, while the general properties and reactions of the members of each family so closely resemble each other that a study of one will give a full clue to the properties of all the other members of the family.

It may be convenient and not without some value if the special features of each of these groups of hydrocarbons are pointed out.

#### FAMILIES OF HYDROCARBONS.

1.  $C_nH_{2n+2}$  series. *Paraffins*. This is a very complete series, comprising about thirty known members, some of which are gaseous at the ordinary temperature, others solid, but the majority are liquids, more or less volatile. Of this series more will be said later on. They are found in the products derived from the distillation of coal, shale, peat, petroleum, wood, etc.
2.  $C_nH_{2n}$  series. Sub-group *a*. *Olefins*. Also a very complete group of about twenty to twenty-five members, comprising both gases and liquids. They are present in the products of the distillation of coal, shale, petroleum, etc.  
Sub-group *b*. *Paraffenes*. A small and unimportant family.  
Sub-group *c*. *Naphthenes*. A small group of hydrocarbons, specially characteristic of Russian petroleum.  
The two sub-groups *a* and *c* will be dealt with in detail later on.
3.  $C_nH_{2n-2}$  series. Sub-group *a*. *Acetylenes*. A small group found in coal distillates.

- Sub-group *b. Allenes*. A small and unimportant group.
- Sub-group *c. Diallyl*. An unimportant hydrocarbon.
4.  $C_nH_{2n} - 4$  series. Sub-group *a. Valylene*. An unimportant hydrocarbon.
- Sub-group *b. Terpenes*. An important though small group of hydrocarbons, especially characteristic of essential oils from plants.
5.  $C_nH_{2n} - 6$  series. Sub-group *a. Dipropargyl*. An unimportant body.
- Sub-group *b. Benzenes*. A small but very important group of hydrocarbons found chiefly among the distillates from coal tar, and also present in small quantities in American petroleum.
6.  $C_nH_{2n} - 8$  series. The hydrocarbon *Cinnamene* is the only known member of this group.
7.  $C_nH_{2n} - 10$  series. *Phenylacetylene*. Found in coal tar.
8.  $C_nH_{2n} - 12$ . *Naphthalene*. A very important hydrocarbon found in coal tar distillates.
9.  $C_nH_{2n} - 14$ . *Diphenyl*. An unimportant hydrocarbon.
10.  $C_nH_{2n} - 16$  series. *Stilbene*. A coal tar hydrocarbon is the sole representative of this series.
11.  $C_nH_{2n} - 18$  series. *Anthracene*. An important hydrocarbon found in coal tar and shale distillates is the only member of this series at present known.
12.  $C_nH_{2n} - 20$  series. Represented by benzyl naphthalene.
13.  $C_nH_{2n} - 22$ . *Pyrene*, which is present to a large extent in the crude shale oil, is almost the sole representative of this series.
14.  $C_nH_{2n} - 24$  series. *Chrysene*, an hydrocarbon very abundant in crude shale oil, is the only known member of this series.

15.  $C_nH_{2n} - 26$  series. Represented by dinaphthyl.  
 16.  $C_nH_{2n} - 28$  series. Not known at present.  
 17.  $C_nH_{2n} - 30$  series is represented by picene, an hydrocarbon found in Californian petroleum.

Of these series of hydrocarbon groups only three possess much interest to the oil chemist, and these are the groups of the paraffins, the olefins, and the naphthene, because it is these three groups that form the various kinds of hydrocarbon naphthas, burning oils, lubricating oils, and paraffin waxes. To these groups of hydrocarbons it is worth paying some attention.

#### PARAFFIN OR METHANE GROUP OF HYDROCARBONS.

General formula  $C_nH_{2n} + 2$ .

Paraffin.	Chemical Formula.	Specific Gravity.	Boiling Point °C.
Methane, . . . . .	$CH_4$	gas	
Ethane, . . . . .	$C_2H_6$	gas	
Propane, . . . . .	$C_3H_8$	gas	—20
Butane, . . . . .	$C_4H_{10}$	gas	—1
Pentane, . . . . .	$C_5H_{12}$	0·600	37
Hexane, . . . . .	$C_6H_{14}$	0·630	70
Heptane, . . . . .	$C_7H_{16}$	0·712	98
Octane, . . . . .	$C_8H_{18}$	0·730	124
Nonane, . . . . .	$C_9H_{20}$	0·741	136
Decane, . . . . .	$C_{10}H_{22}$	0·757	160
Endecane, . . . . .	$C_{11}H_{24}$	0·765	180
Dodecane, . . . . .	$C_{12}H_{26}$	0·776	196
Tridecane, . . . . .	$C_{13}H_{28}$	0·792	216
Tetradecane, . . . . .	$C_{14}H_{30}$	0·812	236
Pentadecane, . . . . .	$C_{15}H_{32}$	0·830	255
Hexadecane, . . . . .	$C_{16}H_{34}$	0·850	276
Heptadecane, . . . . .	$C_{17}H_{36}$		
Octadecane, . . . . .	$C_{18}H_{38}$		
Endecane, . . . . .	$C_{19}H_{40}$		
Icosane, . . . . .	$C_{20}H_{42}$		
Hennicosane, . . . . .	$C_{21}H_{44}$		
Doicosane, . . . . .	$C_{22}H_{46}$		
Triicosane, . . . . .	$C_{23}H_{46}$		
Triacontane, . . . . .	$C_{30}H_{62}$		



From this table it will be seen that a very complete series of this group of hydrocarbons is known, extending, without a break, from the first member with one atom of carbon in its molecule, to the twenty-third with twenty-three atoms of carbon in its molecule. If the formula of each of the members of this series be examined, it will be seen that one member differs from the one above it, or the one below it, by containing one atom of carbon, and two atoms of hydrogen, more or less as the case may be. This difference of  $\text{CH}_2$  between the members of a group is not peculiar to the paraffins, but extends to all other series or groups of organic compounds, and will be noticed again in connection with the olefins and the naphthenes. If the formula be further examined, it will be found that the proportion of carbon to hydrogen, in each of these bodies, is twice the number of carbon atoms plus two. This fact is expressed in the general formula  $\text{C}_n\text{H}_{2n+2}$  applied to the series of paraffin hydrocarbons. The paraffin hydrocarbons are what are called saturated hydrocarbons, that is, the affinities of the carbon atoms are fully satisfied by the number of hydrogen atoms present, and therefore these compounds cannot take up any more atoms of other elements to form new compounds, as is the case with other series of hydrocarbon compounds. To obtain new bodies from the paraffins, it is necessary to bring them into contact with such elements as chlorine, which have a strong affinity for hydrogen, and will take it out of a compound in which it is present; at the same time an equivalent quantity of the element takes its place. Thus, when methane is treated with chlorine, an atom of hydrogen is eliminated from the methane, chlorine taking its place, and forming what is known as methyl chloride,  $\text{CH}_3\text{Cl}$ , while hydrochloric acid is formed at the same time.

The paraffin series of hydrocarbons has the following general features: The first members of the series are

gaseous at the ordinary temperatures of the air; the next members are liquids of varying degrees of specific gravity and boiling points, both of which it will be observed increase as the complexity of the hydrocarbon molecule increases; the higher members of the series are solid bodies, whose melting points increase with an increase in the number of atoms of carbon and hydrogen they contain.

They are not acted upon by treatment with caustic soda or caustic potash at temperatures below the boiling point of water; melted with solid caustic alkalies the higher members of the series do undergo decomposition. Treated with either dilute acids or strong acids at the ordinary temperatures of the air, the paraffins are not affected. Hot strong hydrochloric acid has little or no action; hot strong sulphuric acid exerts a charring action, while hot strong nitric acid decomposes them to some extent. It was their power of resisting treatment with acids and alkalies that earned for them the name of paraffins from two Latin words—*parum*, without; *affinis*, affinity. They are all perfectly stable bodies, and may be exposed to air without undergoing any change. This property is of considerable value as regards the application of the paraffin hydrocarbons to the lubrication of machinery, and other uses. They are all inflammable bodies, and this feature is taken advantage of, the lower members forming the bulk of the paraffin and petroleum burning oils, while the higher members, under the name of paraffin wax, are employed in the manufacture of candles.

The paraffins are found among the products of the distillation of shale, peat, coal, and are present in all petroleum.

There is one feature of the paraffins, which they possess in common with other series of hydrocarbon compounds, and that is their property of forming several isomeric bodies; thus in petroleum two octanes, two nonanes, and two of other

members of the paraffin hydrocarbons are found. These bodies differ from one another in their boiling points, and in the character of the compounds they yield when subjected to various chemical reactions. Warren was the first to point out the existence of these two series of paraffin hydrocarbons in American petroleum, and he gives the following table of the first few members of the two series:—

Paraffins.	Boiling Point.	Specific Gravity.	Boiling Point.	Specific Gravity.																					
Butane	First Series. <table border="0" style="display: inline-table; vertical-align: middle;"> <tr><td rowspan="5" style="font-size: 3em; vertical-align: middle;">{</td><td>gas</td></tr> <tr><td>30°</td></tr> <tr><td>61°</td></tr> <tr><td>90°</td></tr> <tr><td>119°</td></tr> </table>	{	gas	30°	61°	90°	119°	<table border="0" style="display: inline-table; vertical-align: middle;"> <tr><td>0.640</td></tr> <tr><td>0.676</td></tr> <tr><td>0.718</td></tr> <tr><td>0.738</td></tr> </table>	0.640	0.676	0.718	0.738	Second Series. <table border="0" style="display: inline-table; vertical-align: middle;"> <tr><td rowspan="5" style="font-size: 3em; vertical-align: middle;">{</td><td>8°</td></tr> <tr><td>37°</td></tr> <tr><td>68°</td></tr> <tr><td>98°</td></tr> <tr><td>127°</td></tr> </table>	{	8°	37°	68°	98°	127°	<table border="0" style="display: inline-table; vertical-align: middle;"> <tr><td>0.611</td></tr> <tr><td>0.645</td></tr> <tr><td>0.689</td></tr> <tr><td>0.730</td></tr> <tr><td>0.752</td></tr> </table>	0.611	0.645	0.689	0.730	0.752
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0.689																									
0.730																									
0.752																									
Pentane																									
Hexane																									
Heptane																									
Octane																									

The members of the first series are distinguished as the normal paraffins, while those of the second series are called iso paraffins. It will be noticed that the iso paraffins have a distinctly higher boiling point and specific gravity than the normal paraffins. Since Warren's time chemical research has revealed the presence of a large number of instances of such modifications of organic compounds, and has shown that their production or existence is governed by certain laws, which will be found described in the text-books on Organic Chemistry.

Another series of hydrocarbons, found in paraffin and petroleum oils, is the

## OLEFINS.

This group is characterised by every member of it having the hydrogen and carbon of which it is composed united in the proportion shown in the formula  $\text{CH}_2$ ; it is therefore known as the  $\text{C}_n\text{H}_{2n}$  series.

The number of members known is not so numerous as in the case of the paraffins, although there is reason to

suspect that they do exist, but have not yet been isolated or prepared.

The lowest possible term or member would have the formula  $\text{CH}_2$ , methene or methylene, but from theoretical considerations this cannot exist in the free condition, although we are acquainted with compounds derived from it.

#### THE OLEFINS.

Olefin.	Formula.	Boiling Point.	Specific Gravity.
Ethene or Ethylene, .	$\text{C}_2\text{H}_4$	gas	—
Propene or Propylene, .	$\text{C}_3\text{H}_6$	gas	—
Butene or Butylene, .	$\text{C}_4\text{H}_8$	5°	—
Pentene or Amylene, .	$\text{C}_5\text{H}_{10}$	35°	0.663
Hexene or Hexylene, .	$\text{C}_6\text{H}_{12}$	70°	0.699
Heptene or Heptylene, .	$\text{C}_7\text{H}_{14}$	100°	—
Octene or Octylene, .	$\text{C}_8\text{H}_{16}$	125°	—
Nonene or Nonylene, .	$\text{C}_9\text{H}_{18}$	—	—
Decene or Paramylene, .	$\text{C}_{10}\text{H}_{20}$	—	—
Hexdecene or Cetene, .	$\text{C}_{16}\text{H}_{32}$	275°	—
Cerolene, . . . . .	$\text{C}_{27}\text{H}_{54}$	solid	—
Melene or Melissaene, .	$\text{C}_{30}\text{H}_{60}$	solid	—

The hydrocarbons of this group are characterised by the fact that they will combine with chlorine, bromine, or iodine directly, to form the chlorides, bromides, or iodides respectively, each hydrocarbon taking up two atoms of the halogen element. Thus ethene takes up two atoms of chlorine to form ethene dichloride,  $\text{C}_2\text{H}_4\text{Cl}_2$ , while octene forms with bromine the dibromide,  $\text{C}_8\text{H}_{16}\text{Br}_2$ . These halogen derivatives are oily-looking compounds, and it is from this circumstance, and not from their presence in shale or petroleum oils, that their generic name of olefins is derived, although it happens to be rather appropriate, as the olefins form the larger proportion of Scotch and American lubricating oils.

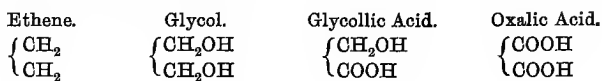
Sulphuric anhydride,  $\text{SO}_3$ , combines directly with the olefins, and in consequence Nordhausen, or fuming sulphuric

acid, readily dissolves them, forming peculiar sulpho-derivatives. Mixed with strong sulphuric acid absorption slowly takes place, and compounds of sulphuric acid with alkyl radicles are formed. Thus ethene forms ethyl sulphuric acid,  $C_2H_5HSO_4$ , from which ethyl alcohol can be got by boiling with water. When liquid olefins of low boiling point are digested for some time with sulphuric acid, they undergo polymerisation, that is, they are converted into higher members of the same series. Thus pentene or amylenes,  $C_5H_{10}$ , which boils at  $35^\circ C.$ , is converted into diamylene,  $C_{10}H_{20}$ , and into triamylenes,  $C_{15}H_{30}$ , which are bodies of higher boiling points and specific gravity. Other olefins are similarly affected. Zinc chloride and other bodies have the property of bringing about this polymerisation. The action of sulphuric acid has some bearing on the treatment of the crude oils in refining Scotch and American lubricating oils. It is customary to treat the crude oils with strong sulphuric acid to remove nitrogenous bodies. If too much acid be employed it is obvious that it may act upon the olefins present and dissolve them, thus leading to a loss of oil.

The olefins have the property of combining with hydriodic acid, hydrobromic acid, and hydrochloric acid to form what are called hydroiodides, hydrobromides, etc. These bodies, when heated with moist silver oxide, give rise to the original olefin and an alcohol of the paraffin series.

The olefins combine with hydroxyl to form a series of alcohols which contain two equivalents of hydroxyl, and are known by the generic name of glycols. Those best known are glycol or ethylene alcohol,  $C_2H_4(OH)_2$ , propylene alcohol,  $C_3H_6(OH)_2$ , butylene alcohol,  $C_4H_8(OH)_2$ . These olefin alcohols can be oxidised, when they yield two series of acids, one monobasic, the other dibasic in character. Both classes of acids are important, and include such well-known bodies as oxalic acid, lactic acid. The relationship between the

olefin and the alcohol and acids derived from it is shown in the following formula :—



Alkalies have no action on the olefins at any temperatures below the boiling point of water, or even slightly above that temperature. They are not prone to oxidation when exposed to the atmosphere.

The larger proportion of Scotch and American lubricating oils are olefins of varying gravities and boiling points.

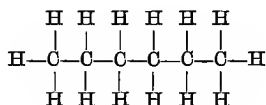
#### NAPHTHENES.

The naphthenes are a group of hydrocarbons which have been found in Russian petroleum oil and in the products of the distillation of rosin. They have not yet been fully investigated, or the true position and relationship of the various members properly ascertained and correlated.

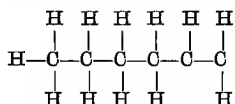
The naphthenes have the general formula  $\text{C}_n\text{H}_{2n}$ , which, it will be observed, is the same as that of the olefins, with which, therefore, they are isomeric, but differ very much in their properties. The first member of the series at present known contains six carbon atoms, and, from various considerations, it seems reasonable to suppose that that is the simplest member which can exist, just as benzene,  $\text{C}_6\text{H}_6$ , is the simplest hydrocarbon of the benzene series.

The naphthenes act like saturated hydrocarbons. Derivatives are known, but they are all obtained by substitution of an hydrogen atom for some other element. By various reactions they can be converted into derivatives of aromatic hydrocarbons of the benzene series. These facts all point to a different grouping of the hydrogen and carbon atoms in the naphthenes, to what is the case with the olefins. In

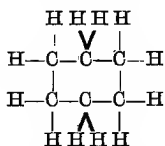
the paraffin and olefin series of hydrocarbons the whole of the reactions which they are capable of undergoing can be readily explained, on the assumption that the atoms are arranged in what is called a chain form, represented by the formula—



for hexane,  $\text{C}_6\text{H}_{14}$ , while the corresponding olefin, hexene,  $\text{C}_6\text{H}_{12}$ , has the formula—



On the other hand, the properties and chemical relationships of the naphthenes can be best explained by assuming that the atoms of carbon and hydrogen are arranged in a ring form, as shown in the formula for six carbon naphthene,  $\text{C}_6\text{H}_{12}$ —



In this all the carbon atoms are connected together in a ring, each with two atoms of hydrogen. An inspection of the two formulæ for the olefin and the naphthene will show how it is that the olefin can form compounds by addition, while the naphthene cannot. In the former case, two of the carbon atoms are connected together with two bonds, one of which can be opened out and combined with chlorine or some other element; on the other hand, in the naphthene, the whole of the bonds of the carbon are engaged.

The other members of the naphthene hydrocarbons are derived from this by substitution of one of the hydrogen atoms by methyl or some other alkyl group.

So far the known naphthenes comprise those which contain from six to fifteen carbon atoms.

The specific gravity of the naphthenes is relatively greater than that of the olefin or paraffin hydrocarbons. Their boiling points are lower, while they are more viscid in character. This can be seen by an examination of the following table, which gives the gravities and boiling points of several members of the three series of hydrocarbons :—

	Sp. Gr.	B. P.
Hexane, $C_6H_{14}$	0.7188	124° C.
Octene, $C_8H_{16}$	0.7294	123
Octonaphthene, $C_8H_{16}$	0.7714	119
Dodecane, $C_{12}H_{26}$	0.7655	202° C.
Dodecene, $C_{12}H_{24}$	0.774	203
Dodecanaphthene, $C_{12}H_{24}$	0.8027	196

These facts explain how it is that Russian petroleum oils have a distinctly lower flashing point than Scotch or American oils of the same specific gravity.

Strong nitric acid acts upon the naphthenes, and converts them into nitro-derivatives of members of the aromatic series of hydrocarbons. Thus octonaphthene yields trinitro-metaxylene, which shows that the naphthene may be regarded as xylene hexahydride. Nonanaphthene yields trinitro-pseudocumene under the same conditions.

Strong sulphuric acid has a stronger action on the naphthenes than it has on the paraffins or olefins; generally sulphonic acids of the benzene hydrocarbons are formed. Chlorine acts on the naphthenes, forming substitution compounds which, on saponification with caustic alkalis, yield alcohols. Bromine yields bromo-derivatives of the benzene hydrocarbons. Thus octonaphthene yields tetra-bromo-



xylene; nonanaphthene, tribromo-pseudo-cumene; and heptanaphthene yields penta-bromo-toluene.

It is therefore evident that the connection between the naphthenes and the benzenes must be very close. Chemists do, in fact, regard the former as hydrides of the latter.

See the *Oil and Colourman's Journal*, November, 1893, to April, 1894, for further information on the paraffins, olefins, and naphthenes, and the chapter on petroleum for list of known naphthenes.

## CHAPTER III.

### SCOTCH SHALE OILS.

THE first hydrocarbon or mineral oils which were known were obtained from a small deposit of what would now be called petroleum oil, found at Alfreton in Derbyshire, which will again be referred to presently. This, however, was soon worked out, and then came oils prepared from what is now known as Scotch shale, a peculiar mineral found only in the district which lies between Glasgow and Edinburgh in Scotland, none being found in any other part of Great Britain, while similar shales have been found in but few localities, New South Wales being the most notable.

The Scotch oil shale presents the appearance of a black, somewhat flaky mass of a homogeneous structure, more or less glossy on the surfaces. If the shale contains much oily matter it can be cut, and when held in the flame of a lamp, will burn with a luminous flame; poor shales are stony and slate-like in appearance. They contain from 72 to 83 per cent. of mineral matter; the rest being of a volatile character. When heated they do not soften or melt, but retain their original form and size; a feature which is of great importance, and is of value in the treatment of the shale.

The principal shale mines lie in the two counties of Edinburgh and Linlithgow. The beds of shale are found interbedded with calciferous sandstone and underlying the lower coal measures. The principal seams of shale are the following:—

The Raeburn shale, which is  $2\frac{1}{2}$  to 3 feet thick, and is worked to a small extent.

Next comes the Mungleshale, which is about 30 inches thick.

About 24 fathoms below this is what is known as the two feet coal, which is not worked; the upper portions of this are good shale.

The Grey shale lies 34 to 50 fathoms below the two feet coal, and averages 34 inches thick. This seam is worked in West Calder, and yields a crude oil notable for the large proportion of solid paraffin wax it contains.

The Houston coal lies 10 to 14 fathoms below the Grey shale. It is mined by many of the oil companies, but without any satisfactory results.

The Fells shale lies from 30 to 40 fathoms below the coal. This is one of the best and most important of the oil shales, and is largely worked; the upper portions of the shale yield as much as 36 gallons of crude oil per ton of shale, while the bottom portions yield 18 gallons, being the richest of the shales in its oil-yielding power. From 20 to 30 lbs. of ammonia sulphate is obtained from a ton of this shale.

The Broxbourn shale. This is one of the thickest seams of all the oil shales, averaging  $5\frac{1}{2}$  feet thick, and is extensively worked. It yields from 16 to 20 gallons per ton of shale, with 40 lbs. of ammonia sulphate, from some portions, while others yield 35 gallons of crude oil and 25 lbs. of ammonia sulphate per ton of shale.

Dunnet's shale lies from 45 to 70 fathoms below the Broxbourn shale, and ranges from 6 to 7 feet thick. It yields from 15 to 30 gallons of crude oil, and 20 to 40 lbs. of ammonia sulphate per ton of shale.

Barracks shale. This has been but little worked; it is of great thickness, averaging 10 feet.

Below this seam comes a series of calciferous sandstone beds, followed by what is known as the Camps limestone at a distance of 100 fathoms below the Barracks shale, while at 120 fathoms below the limestone there is the

Pumpherston shales, which consist of five seams known respectively as the Jubilee shale, about 8 feet thick; the Maybrick shale, 6 feet thick; the Curly shale,  $6\frac{1}{2}$  feet thick; the Plain shale,  $8\frac{1}{2}$  feet thick; and the Wee shale,  $4\frac{1}{2}$  feet thick. These shales yield on an average 20 gallons of crude oil, and from 60 to 70 lbs. of ammonia sulphate per ton of shale.

Before proceeding to describe the process of extracting oil, etc., from these shales, it will be convenient, and lead to a better understanding of the details of the process, if an outline description at first be given.

The shale is mined and then placed in large, vertical iron retorts, heated by fire or hot gases. In the modern retorts steam is also sent into the retorts. As a result, the shale gives off a large proportion of volatile matters, which are condensed, while the mineral or inorganic portion of the shale remains behind in the retort, and is known as "spent shale".

The volatile portions consist of uncondensable gas, which is utilised in the works as an illuminant and as a fuel; water which contains a large proportion of ammonia, and is converted or used for making ammonia sulphate, a valuable manure. Lastly, there is the principal product, a thick, brown, tarry mass, known as the "crude oil".

The crude oil is subjected to a number of operations with the object of converting it into useful products. First it is placed in a still or retort and distilled, when it yields "coke" and "once run oil". This latter product is next treated with sulphuric acid, which extracts from it a large quantity of basic bodies, and for the oil refiner useless hydrocarbon bodies. The treated oil is next treated with a solution of caustic soda, which extracts from it acid bodies; the purified oil is again distilled, and now it is fractionated into three products: a light spirit of a specific gravity of about 0.760, known as "green naphtha"; a light oil, known as "twice run light oil"; and a dark-coloured pasty oil, known as "green oil," while coke remains behind in the still.

The naphtha is purified by treating it with sulphuric acid and caustic soda and redistillation, when "naphtha" is obtained. The twice run light oil is treated with acid and soda, and distilled; it is separated into several fractions which, when further purified, form the various grades of burning oils. The green oil is subjected to a refrigerating operation, which causes the paraffin wax it contains to crystallise out, so that by pressing it can be separated from the oil, forming in its crude condition "paraffin scale," which, when further purified, is converted into "paraffin wax". The oil which is separated from the green oil is known as "blue oil"; it is treated with acid and soda and redistilled into various grades of lubricating oils—light, intermediate, and heavy. The following diagram shows the relationship of the various crude and refined products:—

DIAGRAM OF THE SHALE OIL INDUSTRY.

SHALE. Retorted gives  
 Spent shale.  
 Gas.  
 AMMONIA LIQUOR.  
 Sulphate of ammonia.  
 CRUDE OIL.  
 Coke.  
 ONCE RUN LIGHT OIL.  
 Green Naphtha.  
 Refined Naphthas, 730 to 760 gravity.  
 Twice Run Light Oil.  
 Burning Oils from 810 to 820 gravity.  
 840 Oil.  
 GREEN OIL.  
 SCALE.  
 BLUE OIL.  
 Mineral Colza Oil 840.  
 840 Oil.  
 865 Lubricating Oil.  
 875 Lubricating Oil.  
 885 Lubricating Oil.  
 890-95 Lubricating Oil.

This general scheme is subject to modifications at different works.

The history of the Scottish shale oil industry may be briefly summarised as follows: In December, 1847, Dr. Lyon (now Sir Lyon) Playfair called James Young's attention to the existence of a deposit of oily material at a coal mine at Alfreton in Derbyshire. Young took up the matter, and found that the oil, when purified by distillation, yielded an oil that could be employed in the lubrication of machinery. The crude material was soon exhausted. Then Young turned his attention to Scotland, and found at Torbane Hill a deposit of shale which, when distilled, gave oil. The use of this and the method of extracting oil from it he patented (English Patent, No. 13,292, 1850) in 1850, from which date the Scottish oil trade may be said to have existed. The Torbane Hill mineral was exceptionally rich in volatile matter, yielding at the rate of 120 gallons of crude oil per ton, showing it to contain about 70 per cent. of mineral matter and 33 per cent. of volatile matter. This mineral was exhausted by the year 1862.

Since 1862 the oil has been got from the shale which is found in fairly large deposits in the district which lies between Glasgow and Edinburgh. The various seams of this shale have been already described. Naturally, in the course of nearly half a century, the industry has seen many changes, and many improvements have been made in the details of the process of extraction and in the plant employed. In the early days there was no competitor, but in 1864 petroleum was discovered in America, and in the period from 1864 to 1872 its use was gradually developing, and it was becoming a great competitor of shale oil; and since 1873 the Scotch shale works have had a hard struggle for existence, although at intervals they have had periods of prosperity. The competition with petroleum has had great influence in perfecting the plant and process, and causing the oil works to turn their attention to sundry bye-products, the sulphate of

ammonia and the wax, in which the petroleum works could not compete.

There are three distinct periods in the history of the shale oil trade. The first period extends from 1850 to 1867. The shale was heated in vertical retorts to a strong red heat, so that a large number of charges might be run through, although it was considered that a "low red heat" was essential to the success of the operation. The crude oil obtained was black, tarry, and contained about 8 per cent. of solid paraffin. Little or no attempt was made to utilise the ammonia liquor.

The second period was inaugurated by the introduction of William Young's retort (English Patent, No. 650, 1867) in 1867, and lasted until about 1880. The low red heat was fairly successfully attained, with the result that the crude oil obtained was of better quality, yielding 10 to 11 per cent. of solid paraffin wax. The process of extraction was much improved by the introduction in 1873 of the Henderson retort. Much attention was paid to the bye-products of the process.

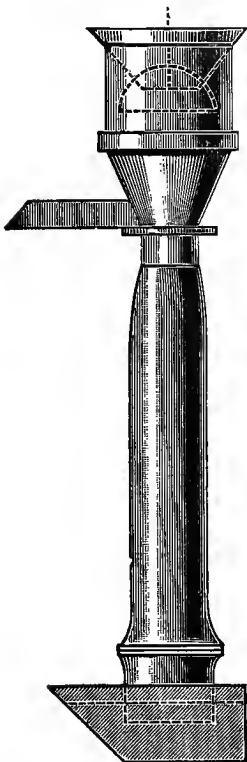
In 1881 was introduced the third period in the retorting of the shale by the introduction of the Young and Beilby retort. In this the low heat was fully attained. The shale was subjected to a double distillation process at first to a low red heat for the purpose of obtaining the volatile products, and, secondly, to the action of superheated steam, by which means the nitrogen contained in the shale was driven off as ammonia. More attention was paid to the purifying of the products, so that a greater yield of refined products was obtained from the shale. The principle first embodied in the Young and Beilby retort of subjecting the shale to a double distillation has been followed up by other inventors with more or less success. Henderson has lately devised a retort which is a material improvement on any hitherto introduced.

Besides the improvements which have been made in the retorting of the shale, the processes and plant employed

in the refining of the crude oils and intermediate oils have been the subject of invention, so that now the finished products are of far better quality, the burning oils are brighter, freer from odour, and burn better; the lubricating oils are brighter, contain less paraffin in solution, while their viscosity and flash points are better; the wax is of higher melting point, and is better in quality, while there is a greater yield of valuable products from the crude oil.

#### PRODUCTION OF CRUDE OIL.

While some attention will be paid to the older methods of retorting and dealing with the shale and crude oil, as being of some slight interest, more notice will be taken of the plant in use at the oil works of to-day.



The earliest retorts employed in the shale oil industry were copied from those used in the coal gas industry, and were of a  $\square$  shape and built horizontally in the furnace. They were made of thin cast iron, and measured 8 to 10 feet long by 35 to 40 inches wide. A number of these retorts were placed in a single furnace and heated by one fire. The weight of each charge was 450 lbs., and usually three such charges were worked off in twenty-four hours. These horizontal retorts soon gave place to what are now known as the old vertical retorts.

The vertical form of shale retort shown in Figure 1 has been the accepted form from very early times in the history of shale



distilling. Figure 2 is a drawing of a set of vertical retorts such as have been in use at the works of the Young's Paraffin Oil Co. at Addiewell. These retorts were 10 feet

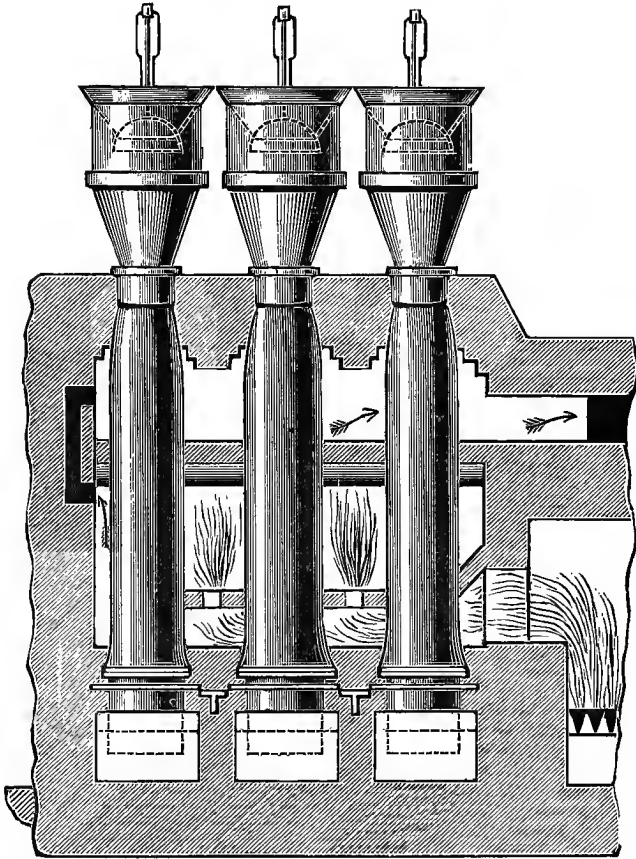


FIG. 2. Bench of Shale Retorts.

long by 2 feet in diameter. Sometimes they were made round, at other times oval, in section. The top of the retorts was made with a hopper valve for the purpose of charging them, the construction of this hopper valve being shown in

the drawing. The bottom was left open, but it dipped into a trough of water which acted as a water seal. The products of the distillation were carried away by a pipe from the top of the retort. They were usually built in sets of six, and were fired from an external fireplace, the flames and heat passing through flues into the space round the retorts. The charge will range from 5 to 8 cwt. The charging and duration of the working were so arranged that 1 ton of shale was worked off from each retort in twenty-four hours. The mode of charging is as follows: The spent shale which lies in the water trough under the retort was raked out by long rakes. This allowed the shale in the retort to fall down, and it in like manner was raked out. Then the valve of the hopper funnel at the top of the retort was opened, and the shale previously placed in the hopper allowed to fall into the retort. These retorts gave a yield of 30 tons of crude oil from 1 ton of shale with 60 to 80 gallons of ammonia liquor, which produced about 15 lbs. of ammonia sulphate. The crude oil was black and tarry, and yielded on refining 5 per cent. of naphtha, 40 per cent. of burning oil, 13 per cent. of lubricating oil, 8 per cent. of wax, and there was 34 per cent. of loss on working up the crude oil. Some of this loss, however, is gas and coke, which is used for fuel and illuminating purposes.

A number of modifications of these old vertical retorts were in use, differing from one another in the shape and size of the retort. Some were long and narrow, others were of large diameter. Then differences were made in the manner of arranging them in the furnace, and in the construction of the heating arrangements. In no case was steam applied to any of the old vertical retorts as a regular method of working.

The first great improvement in the retorting of shale was brought about by the introduction of the Young retort in 1867 (English Patent, No. 650, 1867). These retorts were

built in sets of four. They were double-walled retorts, as may be seen by inspection of the drawing. The outer wall came in contact with the furnace gases and the heat, while the inner retort, which was separated from the outer one by about  $1\frac{1}{2}$  to 2 inches, held the shale. The products of the distillation passed out through an outlet at the bottom, a perforated partition across it preventing any of the shale from getting and choking up this outlet. A similar but larger outlet on the opposite side of the retort served to discharge the spent shale.

A hopper at the top served to feed the retort. A current of gas or steam was sent into the space between the two retorts, and passing through the shale in the inner retort helped to carry off the vapours which were produced, and in the case of the steam helped to increase the proportion of the ammonia liquor produced.

As a comparison of the results which were obtained, the Oakbank shale gave in the old vertical retorts 61.92 per cent. of finished products from the crude oil, of which 8.12 per cent. was paraffin wax. In the new Young retorts the crude oil gave 69.01 per cent. of finished products, of which 11.76 was paraffin wax.

This retort was not long in use, and has been superseded by the Henderson and the Young and Beilby retorts.

The Henderson retort, a sectional drawing of which is given in Figure 3, was brought out in 1873 (English Patent, No. 1327, 1873). It was a great improvement on any that had previously been invented. The Henderson retorts are built in benches of four, are made of cast iron, 15 feet long and about 2 feet in diameter and  $1\frac{1}{4}$  inches thick. At the top is an arrangement for filling the retorts; the products of distillation were carried away by a tubulure at the bottom. The bottom of the retort was formed by a kind of door which opened into the fireplace. The retort chamber is

separate, and divided from the furnace chamber by means of a brick partition, from the centre of which a flue com-

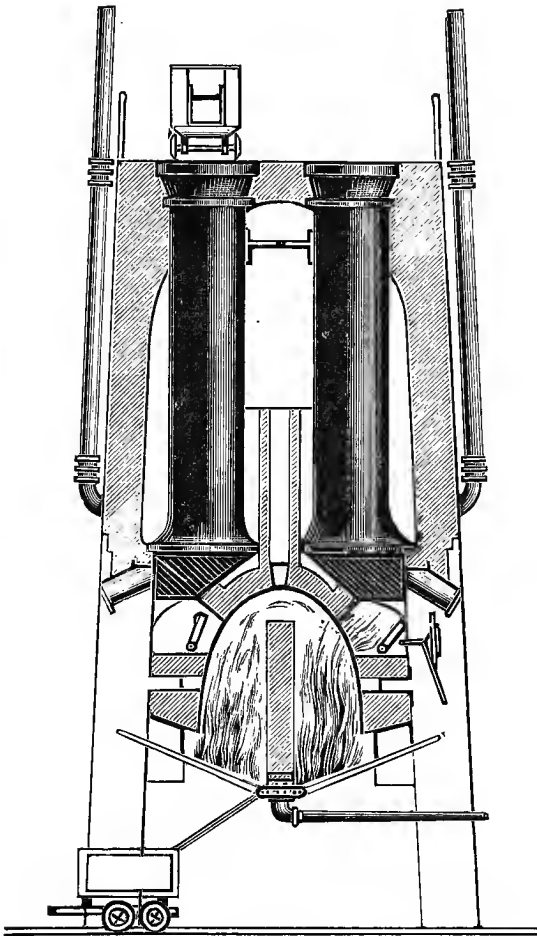


FIG. 3. Henderson Shale Retort.

municated with the retort chamber. By this means the temperature of distillation was kept low, and, further, the

greatest heat was at the top, where the shale first comes into contact with it. The fuel to fire these retorts is partly obtained from the spent shale, which contains a small proportion of carbonaceous matter, and which is thrown into the fireplace when discharged from the retort. The gas which is always produced during the distillation of the shale is also employed for fuel, and these are supplemented by coal or coke.

The charge of a Henderson retort weighs 18 cwt., and takes sixteen hours to work through, at the end of which time the door of the retort is opened, and the spent shale allowed to drop into the furnace, where the carbonaceous matter it contains burns off and serves to heat the retorts. The working of a bench of retorts is so arranged that a retort is emptied and refilled every four hours.

The products of distillation of this retort pass out at the bottom and into the usual hydraulic main that runs along the front of each bench of retorts.

The crude oil obtained from the Henderson retort is lighter in specific gravity, the difference being from 0·02 to 0·03; and it contains a larger proportion of valuable constituents, as will be seen from the following figures:—

	Crude Oil from Henderson Retort.	Crude Oil from Old Retorts.
Naphtha . . . . .	5·00 per cent.	5·0 per cent.
Burning Oils . . . . .	35·00 „ „	40·0 „ „
Lubricating Oils . . . . .	18·00 „ „	13·0 „ „
Scale . . . . .	10·50 „ „	8·0 „ „
Loss . . . . .	31·50 „ „	34·0 „ „

The loss in working is less, while the yield of valuable products is greater.

One hundred tons of shale, when distilled in the Henderson retort, yield 12 tons of crude oil, 8 tons of ammonia water, 4 tons of gas, 67 tons of ash, while in the shale as it

drops into the fireplace there is about 9 tons of combustible matter.

The yield of products from the crude oil has already been given. The ammonia water from 1 ton of shale contains sufficient ammonia to make 16 lbs. of sulphate of ammonia on the average. The gas averages 2000 cubic feet per ton of shale, and it, along with the spent shale, has usually been found sufficient in quantity to fire the furnaces, so that little additional fuel is required.

About the year 1881 some attention began to be paid to the question of increasing the production of the ammonia sulphate from the ammonia water. The demand for ammonia sulphate for manurial purposes having largely increased during previous years, investigations which were carried on by various chemists showed that, on the average, shale contains 0.72 per cent. of nitrogen; and that out of 100 parts of nitrogen present in shale 17 was given off in the form of ammonia, 20.4 per cent. came off in the form of bases in the crude oil, while no less than 62.6 remains behind in the spent shale. It will thus be seen that most of the nitrogen remains behind in the shale. That which comes off in the form of bases, like pyridine, coridine, etc., is practically useless. Whether it can be decreased or not is uncertain, but it would be of decided advantage if that could be done, inasmuch as then the trouble and cost of refining the crude oil would be reduced, and moreover it is probable that the yield of scale and oil would be increased.

Many attempts have been made to increase the yield of ammonia from shale. The addition of lime and soda has been tried, but without any advantage. Hydrogen has been sent into the retorts towards the end of the distillation, with the result that the amount of ammonia has been materially increased, but the cost of the process, compared with the increase in yield, was too high. The introduction of air into

the retorts materially increases the yield of ammonia; but the best material to use is steam, for this not only increases the yield of ammonia, but also that of the paraffin scale.

The amount of nitrogen in shale should, if all came off as

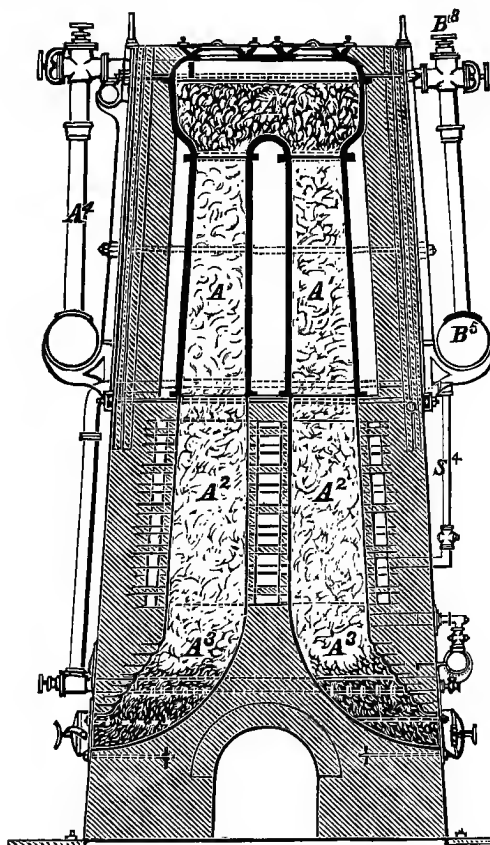


FIG. 4. Young and Beilby Shale Retort.

ammonia, give from 70 to 80 lbs. of ammonia sulphate per ton, but, as a matter of fact, only some 17 to 20 lbs. is usually obtained from the old retorts.

In 1881 Messrs. Young and Beilby, after long experimenting on this subject, introduced a new form of retort

(English Patents, Nos. 1377, 4284, and 5084, of 1882), one object of which was to increase the yield of ammonia. This retort is shown in section in Figure 4, and it is worked in benches along with a gas producer, which is shown

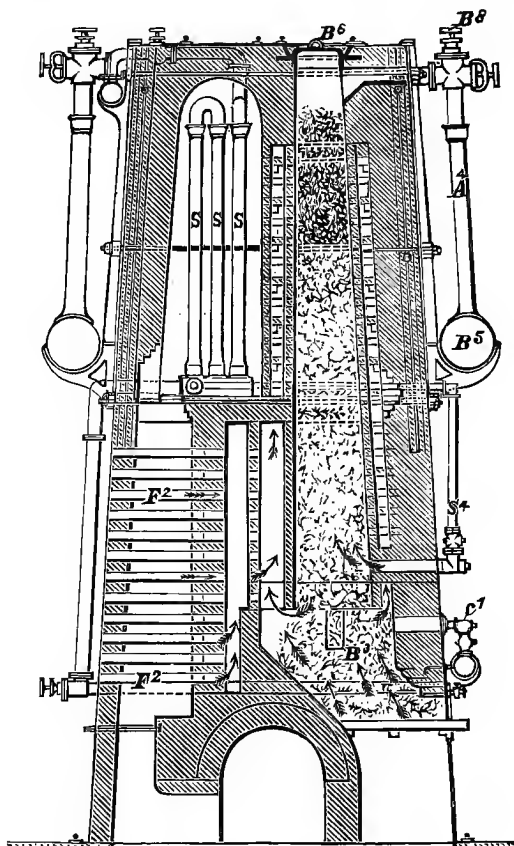


FIG. 5. Young and Beilby Gas Producer.

in Figure 5, and is very similar in construction to the retort itself. This gas producer is necessary, inasmuch as in the last stages of distillation the shale is heated to a high temperature.



The retorts are built in nests of four, as is the Henderson retort. The lower portion of the retort is built of brickwork, the end being curved as shown, and a discharging door is provided. The upper portion of the retort is made of iron; the upper end of the retort opens out into a chamber common to the nest of four, although a separate charging hopper is provided to each retort. The products of distillation pass off from the top of the retort.

The shale is charged into the hopper, and is heated by hot gases from the retort below. As spent shale is removed from the discharging door at the bottom, the shale gradually moves down and passes through each portion of the retort. In the iron portion of the retort it parts with its hydrocarbon oils and paraffin, while in the brick portion the carbon is more or less completely burned away by the heat and steam with which the shale comes in contact, and water gas and ammonia are produced. In the brick portion of the retort the shale is heated to a white heat, and comes in contact with superheated steam, the effect of which, according to Grouven (*Versuchsstationen Jour.*, 28,343), is that the nitrogen in the shale is converted into ammonia, the steam preventing its after-decomposition, and, moreover, exercising a useful influence on the production of hydrocarbon oils and paraffin in the upper portions of the retort. The retorts are heated by gas; that which is produced in the distillation of the shale is employed along with some specially produced by a gas producer, the construction of which is shown in Figure 5. This gas producer consists of a vertical retort built of brick. At the top it is closed with a charging door, while from the top end passes a pipe carrying off the volatile products which are formed. The lower end of the retort is closed by a fireplace and ashpit, which are fitted with regulating doors and dampers. A number of flues surround the retort, communicating with the bottom of the retort. These gas pro-

ducers, like the shale retorts, are built in nests of four. The producer is fed by coal. In the upper portion it undergoes some distillation, the temperature being a red heat, the gases and vapours which are formed passing away by the exit pipe that is provided, and cooled and collected. The coke which is formed gradually passes down the retort and comes into contact with a current of steam, and now it is decomposed, water gas and ammonia being formed. These pass away with other volatile products, the ammonia being collected and the gas used for heating the retorts. Some of the coke escapes the decomposing action of the steam. This when it comes down to the firebars is burnt to carbon monoxide, and this passing into the flues burns and serves to heat the retort or producer.

These gas producers yield from 90 to 120 lbs. of ammonia per ton of the coal used, which contains an amount of nitrogen equal to 165 to 170 lbs. per ton, so that a large proportionate yield is obtained. The gas which comes from the producer consists of carbon dioxide, carbon monoxide, methane, hydrogen, and nitrogen, hydrogen being the most important, usually forming 28 per cent. of the gas. Nitrogen exists to the extent of 44 per cent., while the carbon dioxide forms about 10 per cent. The composition of the gas will, however, vary from time to time.

The Young and Beilby retorts are worked with steam and air being sent into them. The introduction of these bodies has a material influence on the amount of ammonia and paraffin obtained. It has been ascertained that, working without steam and air, 1 ton of shale will give about 30 gallons of crude oil, containing  $10\frac{1}{2}$  per cent. of solid paraffin. Working with steam and air, the yield of crude oil rose to 33 gallons, and it contained  $12\frac{1}{2}$  per cent. of solid paraffin, while the ammonia was nearly doubled in yield.

Previous to the introduction of these retorts at the Oak-

bank works, the old vertical retorts in use gave from 15 to 16 lbs. of ammonia sulphate per ton of shale, while the new

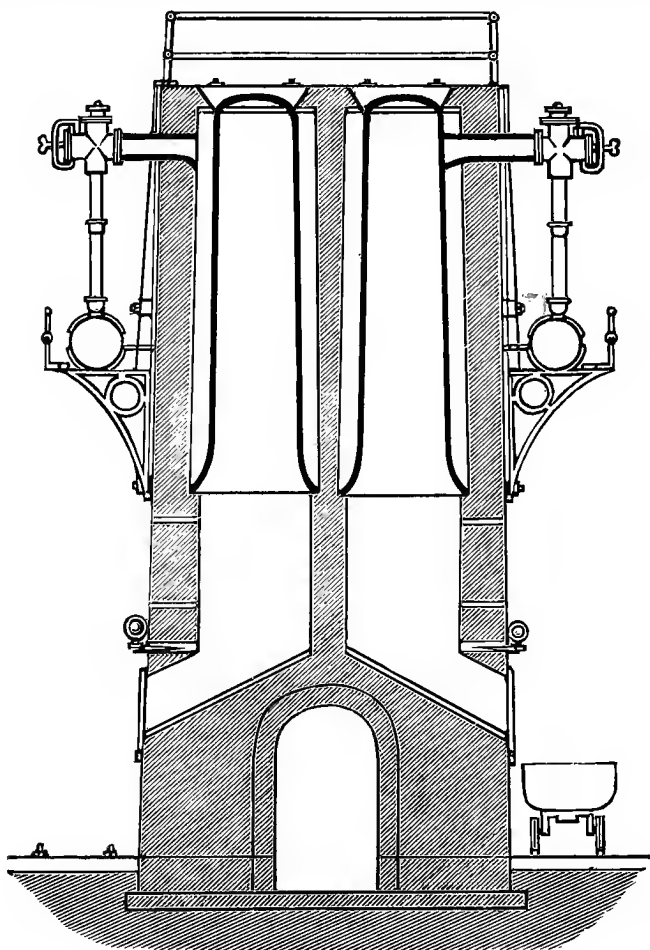


FIG. 6. Couper-Rae Shale Retort.

ones gave 30 lbs. per ton. The crude oil from the Young and Beilby retort is of lighter specific gravity and is far purer in quality, so that it is possible with it to do away

with the first redistillation and begin to fractionally distil it at once, there being thus a saving of expense in the treatment of the oil.

In 1883 the Couper-Rae retort was brought out. This is shown in Figure 6, from which it will be seen that this retort closely resembles the Young and Beilby retort, but the lower portion is built of solid brickwork, while in the Young-Beilby retort it is built open for the flames and heat from the burning gases to play around the retort. Further,

in the Couper-Rae retort a mixture of steam and air is introduced by a steam jet in the lower portion of the retort.

The Stanrigg retort (English Patent, No. 9783, of 1889) was introduced in 1889. It is shown in Figure 7, which shows it to be very simple in form. It is built of brick cased with iron. It is 46 feet high, this height having proved to be the least which will yield the maximum amount of ammonia from the shale. The diameter is  $7\frac{1}{2}$  feet at the top and 11 feet at the bottom. The charging is done at the top, from which end also the

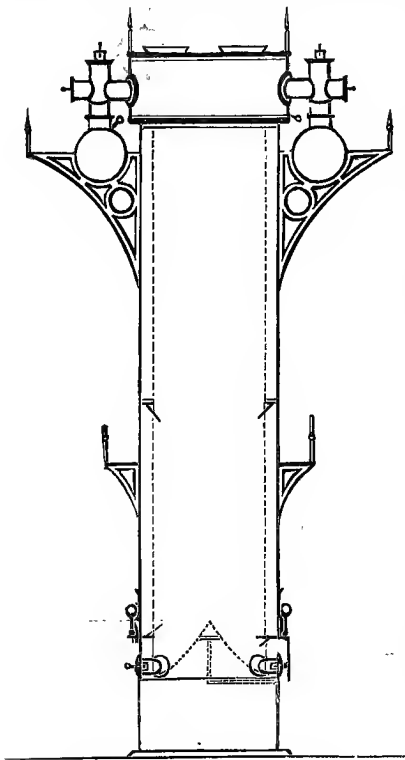


FIG. 7. Stanrigg Shale Retort.

gases and vapours are drawn off, the exit being favoured by using a Roots blower. Low pressure steam, using about

100 lbs. per ton of shale, is passed into the retort at the bottom, while some air is also drawn through the retort, passing

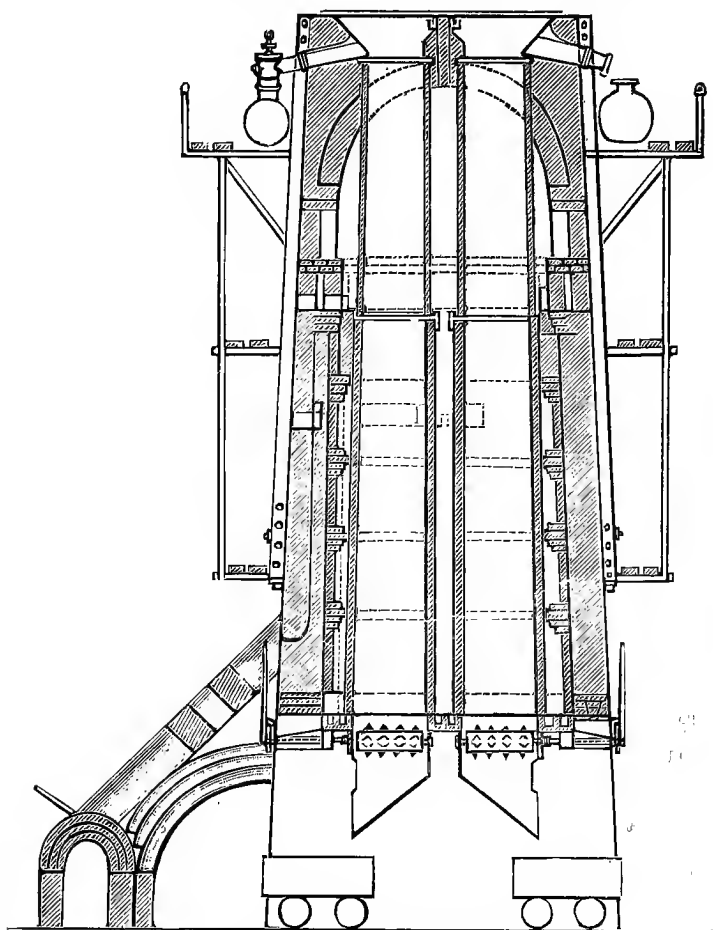


FIG. 8. Henderson Shale Retort.

in at the discharging doors. The charge is 60 tons, and it takes five days to work it off, 12 tons being removed each day and replaced with fresh shale. The heat attained in

this retort is about the least of any, and is well under control. Stanrigg shale distilled in this retort gives 40 gallons of crude oil of a specific gravity of 0·860 per ton, with 30 lbs. per ton of ammonia sulphate. The gas amounts to 60,000 cubic feet. There is but little naphtha yielded in this retort, which is rather disadvantageous, and arises from the large quantity of gas which is evolved, preventing effectual scrubbing.

In 1889 was introduced by Mr. Henderson a new form of retort (English Patent, No. 6726, of 1889), which is shown in Figure 8. This new retort is vertical, and is 28 feet long. The upper portion is made of cast iron, and here the oil is distilled from the shale at a temperature of about 900° F. The lower portion is built of brick, and is maintained at a temperature of about 300° F. The brickwork portion of the retort is surrounded by flues into which the gas from a gas producer is sent. The gas burning in the flues heats the retorts. The bottom of the retorts is formed into a shoot, so that the spent shale can be delivered into waggons running on rails below the retorts. In the bottom of the retort are fitted rollers with teeth, the revolution of which forces out the spent shale. One peculiarity of this retort is that it is being continually discharged, and not in an intermittent manner, as is the case with the other retorts, so that the charge is continually dropping down the retort. This continuous motion is said to increase the yield of ammonia and gases from the shale. Further, that it prevents any fusion of the shale, and therefore removes any liability to aggregate together which sometimes occurs in the older forms of retorts. The products of combustion are drawn off from the top of the furnace.

The following gives the comparison between the working of this new Henderson retort and the old form of 1873:—

	1873 Retort.	1889 Retort.
Crude Oil in gallons per ton . . .	31	31
Sulphate of Ammonia, lbs. per ton	17	44
Gas, cubic feet per ton . . . .	2000	15,000

The great advantage is in the increased yield of ammonia, while at the same time the quality of the crude oil is better.

*The Process of Distillation.*—The shale is thrown into the hopper of the retort, and on opening the valve it drops into the retort. In the majority of retorts this operation is performed at certain regular intervals, the operation of charging being preceded by that of discharging in the case of the old vertical retorts. This latter operation was effected by withdrawing the spent shale through the water seal at the bottom of the retort. This drawing was done at the end of every hour, but the retort was only freshly charged every three hours, about 3 cwts. being run in each time. The Henderson first retort was drawn and charged every four hours, the weight of each charge being 4 cwts. The drawing in this retort is effected by opening a door at the bottom of the retort, whence the spent shale drops into the fireplace below. The Young and Beilby retorts were also charged once in four hours, with about 4 to 5 cwts. of shale each time, the spent shale being first drawn out at the bottom. The Stanrigg retort was only charged about two to four times each day, the daily charge amounting to 12 cwts. In the case of the new Henderson retort, the charging is done at intervals of about two to four hours, but the discharging is done much more frequently by turning the discharging rollers at the bottom, by means of the lever which is provided for that purpose.

The volatile products of the distillation come off in a pretty regular order. First there is hydrocarbon gas, then comes a colourless light oil, then a yellow oil containing solid paraffin, then dark brown oils containing much alkaloidal matters. This is the order when a quantity of shale is being

distilled throughout. Besides these oily bodies, water is found accompanying them. In modern shale distilling retorts the whole charge of shale is at varying stages of its decomposition; that at the top is just giving off its lightest products, that at the bottom is giving off its heaviest products, while the intermediate portions are at intermediate stages of treatment. Therefore, from the exit pipe, there is passing a complex mixture of volatile products.

#### COLLECTION AND TREATMENT OF VOLATILE PRODUCTS FROM SHALE.

The volatile products obtained in the distillation of shale are carried out of the retorts by suitable pipes, generally from the top of the retort, but in one or two forms, as has been previously noted, from the bottom. These exit pipes are in communication with an hydraulic main which passes along the front of the bench of retorts. In this main some condensation of the less volatile products will take place, but the amount is not large; necessarily it varies with every different kind of retort. Owing to the very different temperatures at which the gases and vapours issue from the exit tube, in some this temperature will reach about 1000° F., in others it may not exceed 500° F.

From the hydraulic main the products pass through a series of upright  $\cap$  shaped tubes, standing on a large iron box divided into compartments, so that one leg is in communication with one compartment, and the other leg with the next compartment. These tubes act as condensers, the arrangement of the condensers on the iron compartment box ensuring that the products will pass through the whole series of tubes, and therefore the products will become fairly well condensed. In winter, of course, the condensation is much better than in the hot summer weather, when occasionally it may happen that some of the more volatile products escape condensation.



These are recovered as mentioned below. In the condensers the ammonia water and crude oil condense and collect in the box, from which they flow into separators.

#### SEPARATORS.

The separators are shown in a sectional diagram in Figure 9. They consist of an iron box divided into two

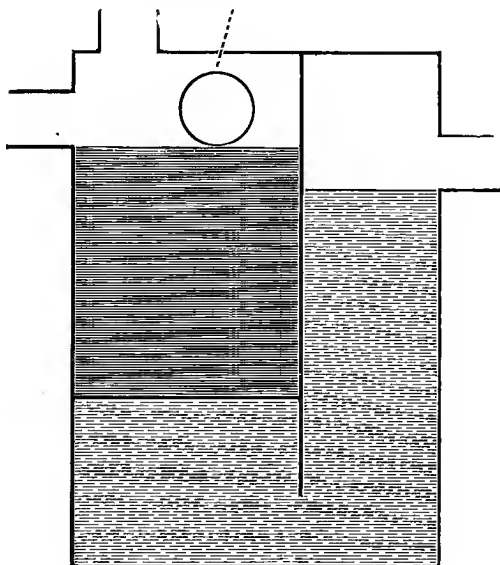


FIG. 9. Crude Oil Separator.

compartments by a partition which does not extend quite to the bottom, and divides the box into two unequal-sized compartments. The inlet pipe for the products from the condensers opens into the larger of the two compartments on the top. From one side of this, near the top, is an opening through which escapes the crude oil; from another side of the same compartment is another opening through which flows the gaseous and uncondensed product; from an

opening in the smaller compartment, which is placed somewhat below the level of the oil opening, flows the aqueous portion of the distillates. The water, being heavier than the oil, sinks down to the bottom in the separator, and, passing under the dividing partition, flows out through the exit pipe provided for it; while the oil, being lighter, remains in the first division of the separator, and flows out through its exit pipe.

*The Gases.*—The uncondensed products which pass through the separators, consisting chiefly of uncondensable gas, but which often contain portions of light hydrocarbon vapours, are next passed through scrubbers. These consist of a tall tower, filled with broken bricks, etc., down which flows a stream of naphtha or light oil, which, exerting a solvent action on the hydrocarbon vapours in the gases, dissolves them, and carries them down to be afterwards recovered. The gases themselves are then sent forward to the retorts to be used for fuel, or they may be used for lighting the works or other purposes to which gas can be put. The amount usually produced is 2000 cubic feet per ton of shale, but the quantity varies with the kind of retort which is used in the operation of distilling the shale.

*The Ammonia Water.*—This usually has an average specific gravity of  $2\frac{1}{2}^{\circ}$  Twaddell (1.012), and contains ammoniacal compounds equivalent to 0.277 lb. ammonium sulphate per gallon. It contains ammonium carbonate, which is the principal compound present, ammonium sulphide, ammonium sulphite, ammonium sulphate, ammonium thiosulphate, all of which are present in traces only. This ammonia water is converted into ammonium sulphate by treatment with sulphuric acid, a plan which was first adopted by Robert Bell, in 1864, at the Broxbourn Works. The ammonia sulphate has been found very valuable as a manure.

## TREATMENT OF THE CRUDE OIL.

The crude oil obtained in the distillation of shale is a most complex mixture of various bodies. A great variety of compounds have been found in it, but it is doubtful whether it has yet been fully investigated, and the whole of its constituents known. It contains:—

1. Paraffins ranging from  $C_4H_{10}$  to  $C_{30}H_{62}$ .
2. Olefins ranging from  $C_4H_8$  to  $C_{20}H_{40}$ .
3. Crotonylenes,  $C_6H_{10}$ ,  $C_7H_{12}$ ,  $C_8H_{14}$ .
4. Hydrocarbons. Benzenes are present in but small quantities, and are absent in some shale oils. The presence of naphthalene and anthracene is doubtful. Pyrene,  $C_{16}H_{10}$ , and chrysene,  $C_{18}H_{12}$ , occur in notable quantity.
5. Nitrogen bases. Ammonia, members of the pyridine series, coridine, rubidine and viridine are present, but no members of the aniline series.
6. Oxygenated bodies. Acids of the acetic series are present; phenols and oxyphenols and thymols are present.
7. Sulphur compounds. These are present in small quantity. Sulphur is present to the extent of 1·5 per cent. in shale. Of this 1·4 is left behind in combination with the spent shale, 0·025 per cent. is found among the gaseous products, 0·028 is found in the crude oil, and 0·02 in the ammonia water. The presence of sulphur in shale leads to the diminution of the amount of paraffin wax, and a low yield of other products.

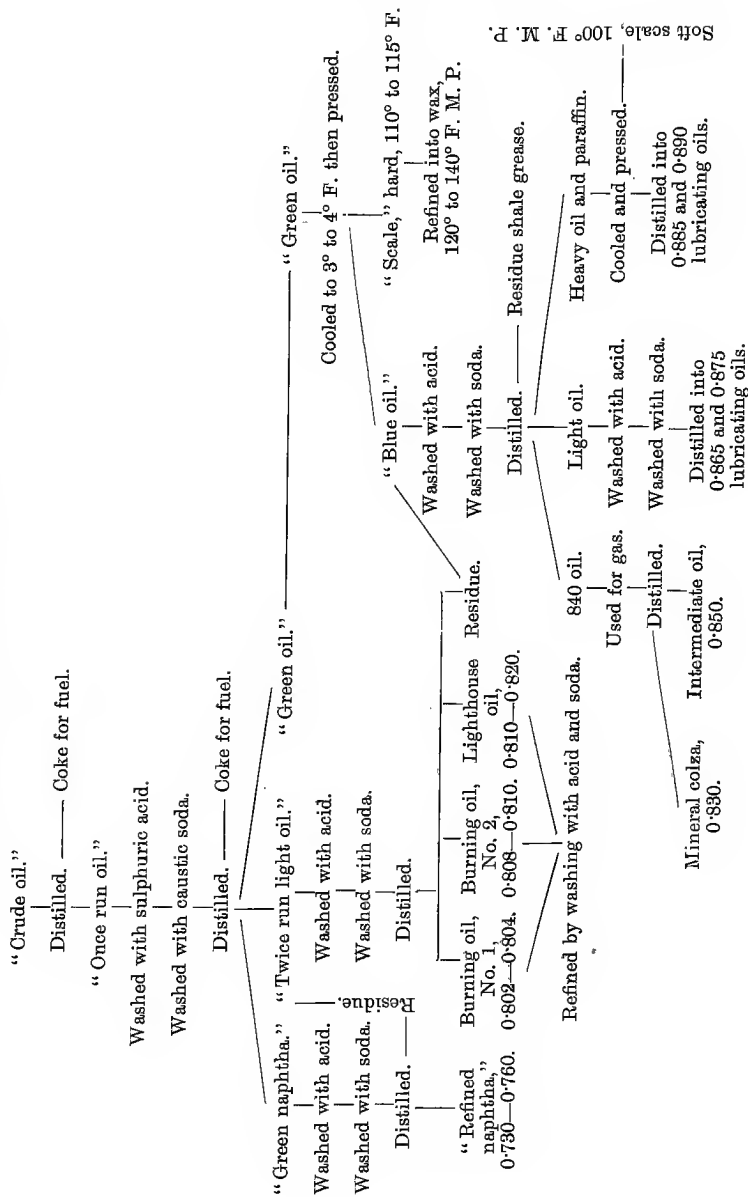
Of all these bodies found in crude oil, only the paraffins and olefins are of any service to the shale oil distiller. The rest are of no use whatever, and have to be got rid of, and their presence increases the difficulty of refining the crude oil.

The principles on which crude oil is refined are: First, that sulphuric acid has a solvent action on the heavier hydrocarbons—naphthalene, anthracene, pyrene, chrysene, etc., and on the nitrogen bases which are present. Second, that caustic soda has a solvent action on the oxygenated and sulphur compounds. Thirdly, that by fractional distillation, the oils can be separated into hydrocarbons of varying gravities and boiling points.

The process of refining crude shale oil, therefore, consists in treatment with sulphuric acid, caustic soda and distillation.

The following diagram gives an epitome of these processes and shows where the various operations come in:—

## DIAGRAM OF THE SCOTCH SHALE OIL INDUSTRY.



This diagram does not pretend to be complete, but simply to give a general idea of the method of working. The details of refining vary at different works; some produce a comparatively small number of products, others carry the refining rather further and produce a greater variety.

CRUDE OIL is a dark, reddish brown, pasty mass, with a peculiar odour. It has a specific gravity of from 0·865 to 0·890, the tendency being with modern improvements to produce a lighter crude oil.

This crude oil is refined into useful products by a process depending largely upon distillation, combined with treatments by sulphuric acid and caustic soda. The distillation of crude oil may be carried out in an intermittent manner, or in a semi-continuous manner, the latter being the most modern idea of refining shale oil.

The intermittent method of refining crude oil is carried out in the following manner: The crude oil is put into a

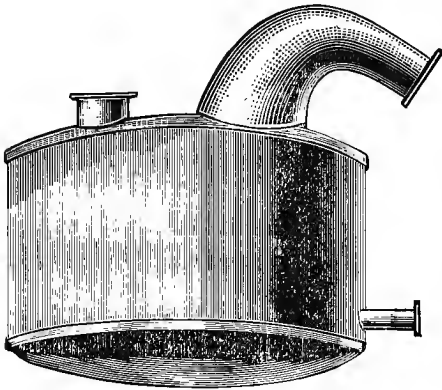


FIG. 10. Shale Oil Still.

large iron still which is cylindrical in shape, a concave bottom and a large manhole on the top. Such still is shown in Figure 10. It may be made 7 feet in diameter and 5 to 7 feet high, and is 1 inch thick. From the top rises a goose-neck, which is in connection with a worm condenser. The still varies in size at different works, and may be constructed to hold from 250 to 2200 gallons of oil. The still is set in a suitable brickwork furnace.

When the still is first set in operation some gas comes over, at about 100° to 120° F.; this is followed by a light colourless oil. When the temperature gets higher, a pale yellow oil comes over; this oil gradually darkens in colour, and soon particles of solid paraffin begin to show themselves. Finally a dark brown oil comes over; this contains the great bulk of the alkaloidal matters of the crude oil. The distillation is carried on until nothing comes over, and a mass of coke is left behind in the retort.

At first the condenser is kept full of cold water; when, however, the oil begins to get of a dark yellow colour, the water is run off; then when solid paraffin shows itself, warm water is run into the condenser to keep the paraffin in a liquid condition or otherwise the pipes might become choked up.

The oily matter which comes over is known as "Once Run Oil". The modern shale retorts, Young and Beilby's and Henderson's, are found to give crude oil of so much greater purity than what the older patterns of retorts did, that it has been found possible to dispense with this first distillation and proceed with the next operation, thus saving a considerable item in cost.

In some cases steam is sent into the still, but this is not customary.

**ONCE RUN OIL.** This has much the same appearance as crude oil, but is rather paler in colour and lighter in specific gravity, which is usually 0·845 to 0·876, and it forms some 90 to 95 per cent. of the original crude oil.

The first treatment to which once run oil is subjected consists in running it into large iron tanks holding about 8000 gallons and fitted with either a mechanical agitator or with an arrangement for agitating with air, the latter plan being considered to be the best. In these tanks it is mixed with from 2½ to 4 per cent. of its volume of strong sulphuric

acid of 1.74 (150° Twaddell), the acid and the oil being thoroughly mixed by the agitator. As regards a mechanical agitator, this usually takes the form of an iron shaft carrying arms which, running round in the oil, agitate it. The air agitator usually takes the form of a copper box placed at the bottom of the tank; the box has perforated sides, and is in communication with a blower which forces air through the mixture of oil and acid. After being agitated together for some time the mass is allowed to stand for about twenty-four hours, when the acid settles to the bottom, carrying with it all the impurities which it has dissolved out of the oil. The layer of acid at the bottom of the tank is then run off, leaving the purified oil. This acid layer has a black colour and a strong odour of sulphur dioxide; it is known as the acid tar.

The acid has no action upon the paraffins present in the once run oil, and comparatively little if any on the olefins. Some of the other hydrocarbons it polymerises, and the crotonylenes which are present are probably converted into acetylenes, for those bodies have been found present in the acid tar while they are absent in the original oil. The pyrene and chrysene are dissolved, while the nitrogenous bases are combined with the oil and become dissolved therein. It will thus be seen that the sulphuric acid has a great purifying action on the oil. It is not advisable to use too much or too strong sulphuric acid, or it may attack some of the higher members of the olefin series of hydrocarbons which may be present, and this is not desirable, as these bodies are useful lubricating agents.

The acid tar which is obtained at several stages of the process of refining shale oil (see Diagram, page 51) is mixed with water, which causes the oil to separate; this is collected and used as a fuel, being burnt by means of a spray burner. The acid is employed in the manufacture of ammonia sulphate.



The second treatment of the oil consists in running into the oil in the tank from  $2\frac{1}{2}$  to 4 per cent. of its volume of a solution of caustic soda, of a specific gravity of 1.30 (60° Twaddell), thoroughly agitating the oil and alkali together and then allowing them to stand to settle. The alkali goes to the bottom and carries with it all the acid and sulphur constituents of the oil, the alkaline mixture which is thus obtained being run off and forming what is known as "soda tar". The washed oil is then ready for the next operation.

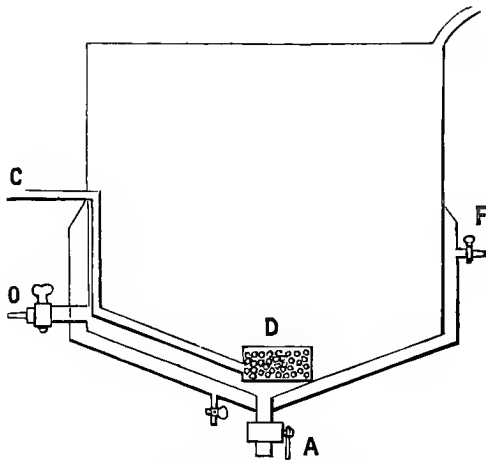


FIG. 11. Oil Washing Tank.

Figure 11 shows one form of tank in which the treatment with acid and alkali can take place. The lower portion of this tank has a steam jacket, so that the contents may be heated to about 150° F. to ensure their being perfectly fluid. It is fitted with an air agitator D, in connection with an air pipe C, an outlet for the tar A, an outlet for the washed oil O, and a steam pipe F to jacket.

The soda tar is treated by blowing air into it, which causes the formation of carbonate of soda and the liberation

of oily matters dissolved in it ; these are known as creosote, but have little use. The carbonate of soda can be recausticised for future use.

The WASHED ONCE RUN OIL is now run into a still of some 2000 gallons capacity. This still differs somewhat in form from that used in the first distillation of the crude oil, and is shown in Figure 12. Inasmuch as in this second distillation the operation is not carried out until a

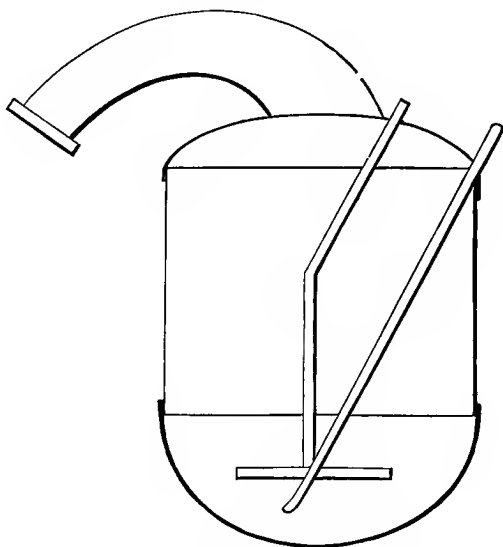


FIG. 12. Oil Still.

dry residue is left behind, it is found better to employ a still (Figure 12) with a convex bottom, so that when the distillation is ended the residual liquid can be blown out through a pipe provided for that purpose. In this second distillation the oil is subjected to a fractional separation. As the distillation proceeds, the distillates are tested from time to time. When the specific gravity of it reaches 0.760, the receiver is changed, or rather the direction of the stream of distilled

oil is changed, and run into another tank. What has come over is "green naphtha," the refining of which will be dealt with presently. Then the distillation is carried on until evidences of solid paraffin begin to show themselves in the condenser, and then the direction of the stream of distillates is again changed. What has come over is known as "twice run light oil," and it is subsequently refined into burning oils of various qualities. The distillation is then proceeded with until a dark brown oil comes over, and but little, about 5 per cent., of the original oil is left in the still, then it is stopped. What has come off is known as "green oil," and is afterwards refined into lubricating oils and solid paraffin wax. The residue in the retort is used for making grease, fuel, etc., and is known as "still bottoms," "shale grease".

This system of distilling the crude shale oils is rather costly. The wear and tear on the stills by being heated to redness in the first case to run the oil to coking point, and the breaking up of the furnaces by the unequal expansion due to the cooling and heating which must occur in the intermittent system of working, cause a very large outlay for repairs. Still with the older forms of retorts it was necessary, inasmuch as the crude oil was not pure enough to fractionally distil at once, and there is no doubt but that, during the first distillation, some of the heavier hydrocarbons were broken up with the formation of lighter hydrocarbons, a feature which has been observed in the treatment of petroleum, and has been termed "cracking". The more modern retorts, however, give a much purer crude oil, which may at once be fractionally distilled. There has thus been devised a system of continuous or semi-continuous distillation, principally by Mr. Henderson of the Broxbourn works, where it is carried out to its greatest length.

The SEMI-CONTINUOUS SYSTEM of distilling the shale oil is carried out in a set of five stills. These are shown in Figures 13 and 14, which are taken from a paper read by Mr. D. R. Stuart, and published in the *Journal of the Society of Chemical Industry* for February, 1889. In this method of distilling two kinds of stills are employed. First there is a set of three boiler-shaped stills set in a suitable brickwork, and heated by separate fireplaces. These three boilers are, for the sake of economy, set in a single setting. Besides these there are two coking stills of the form shown in Figure 14. The crude or once run oil is run into the centre one of the three boiler-shaped stills, and it is heated. When the naphtha begins to distil off, the supply of crude oil is allowed to run in at a rate equivalent to the rate at which the distillation proceeds. This still is therefore kept at one uniform heat to permit of this being done. The oil from which the naphtha has been distilled off is run into one of the end stills and is heated to a higher temperature, and from it the burning oils having a gravity of 0.835 are distilled off. From this still the oil runs into one of the coking stills to be run dry. What comes over in this still is the green oil. It will be understood that there is a constant flow of crude oil in at one end of the central still, and distilled oil out at the other end into the second still. When this is full the flow of oil is transferred to the third still at the other end of the set. It is found that one naphtha still will keep two burning oil stills in work, and for these two coking stills are required.

The main advantage of such a system of distilling is that a greater volume of oil can be treated in a given time, that the regular heat at which they are maintained greatly reduces the wear and tear on them, and so reduces the cost of repairs. A further economy is effected by causing the oils fed into the stills to be heated by the hot vapours which are passing off from the stills themselves.

Three to four products are obtained in distilling fractionally once run or crude oil by either of the methods described.

1st. Green naphtha.

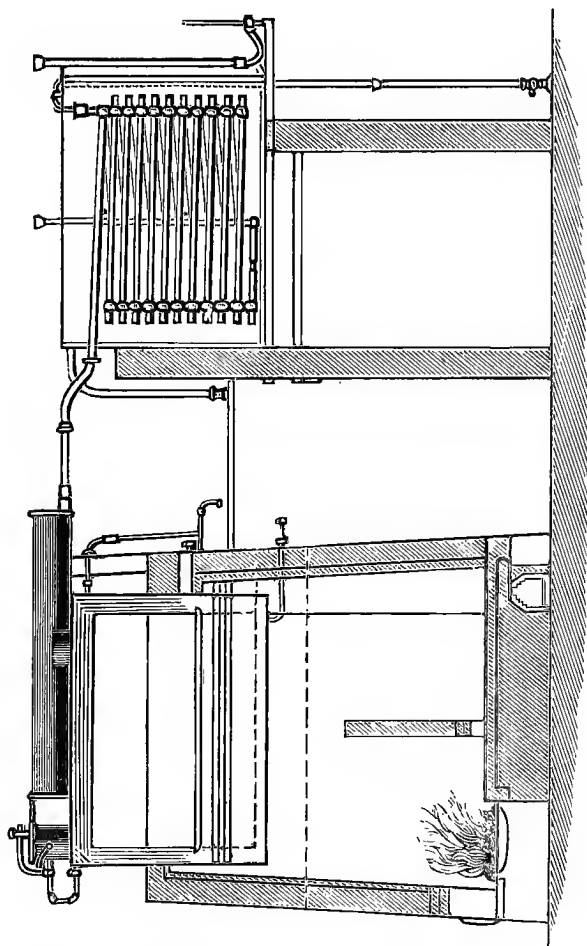


FIG. 13. Continuous Oil Still.

2nd. Twice run light oil.

3rd. Green oil.

4th. Coke or shale grease.

These products are now subjected to various refining

operations before they are sent out as commercial products.

The *green naphtha* is first treated with 1 to  $1\frac{1}{2}$  per cent. of its volume of sulphuric acid, then with  $1\frac{1}{2}$  to 2 per cent. of its volume of caustic soda, these operations being called washing; the tars which are in each case produced being added to the corresponding tars obtained from the crude oil or once run oil, and treated with them.

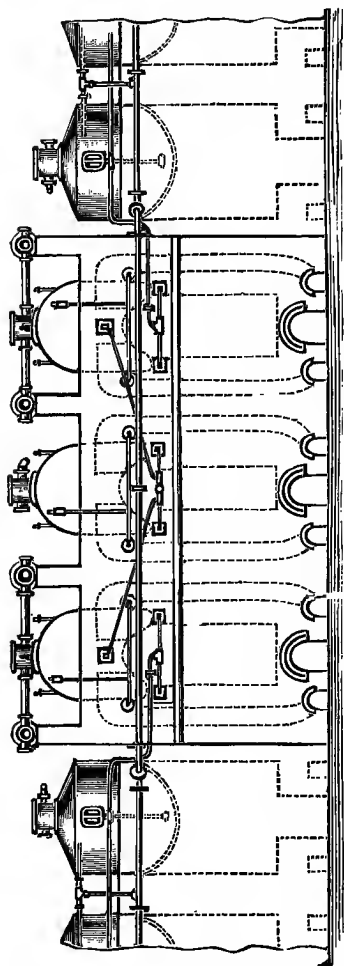


Fig. 14. Continuous Oil Stills.

The washed naphtha is next put into a still and distilled until the distillate has a specific gravity of 0.760. Sometimes two fractions are collected, a light naphtha having a gravity of 0.730, and a heavy naphtha having a gravity of 0.760. Whatever remains in the still after this naphtha has come off is put into the twice run light oil, and refined along with that.

*Shale naphtha* is a water-white, very limpid oil or spirit. Its specific gravity varies from 0.730 to 0.760. It is quite insoluble in water, and will not mix with that fluid. It is inflammable, and on the application of a light will take fire, burning

with a luminous flame. It is therefore highly important when working with naphtha to be careful that no light can come in contact with it or its vapour. It is very volatile, and on exposure to the atmosphere evaporates completely away without leaving any residue behind. It is not acted upon by caustic soda, sulphuric acid, or nitric acid, at all ordinary temperatures, or even when heated to a moderate degree. Long digestion with strong sulphuric acid will cause the olefins it contains to be dissolved in the acid, but the paraffins are untouched. Naphtha is a powerful solvent of oils, fats, and similar bodies. It does not dissolve resins freely, the most soluble being dammar. It is employed as a solvent in the preparation of paints and varnishes, and in other industries.

Naphtha begins to boil at about  $60^{\circ}$  C., sometimes even higher; usually from 4 to 5 per cent. distils over below  $100^{\circ}$  C., 10 per cent. under  $125^{\circ}$  C., 60 per cent. under  $160^{\circ}$  C., and 90 per cent. under  $190^{\circ}$  C. This is the usual average rate of distillation for a heavy naphtha. A light naphtha will distil at somewhat lower temperatures.

Shale naphtha is a complex mixture of hydrocarbons which principally belong to two series, paraffins and olefins, the latter forming about 60 per cent. of the naphtha. There has been obtained—Hexene,  $C_6H_{12}$ , heptene,  $C_7H_{14}$ , octene,  $C_8H_{16}$ , and nonene,  $C_9H_{18}$ , among the olefins; and hexane,  $C_6H_{14}$ , heptane,  $C_7H_{16}$ , octane,  $C_8H_{18}$ , and nonane,  $C_9H_{20}$ , among the paraffins, but other members of these hydrocarbons are present.

The TWICE RUN LIGHT OIL is refined by washing with 1 to  $1\frac{1}{2}$  per cent. of sulphuric acid, and 1 to  $1\frac{1}{2}$  per cent. of caustic soda, as has been previously described. It is then run into stills, and usually fractionated into three fractions:—

1st. Having a gravity of from 0.800 to 0.810.

2nd. Having a gravity of 0.810 to 0.820:

3rd. Having a gravity of 0·820 to 0·830.

What is left in the still, after running off these fractions, is added to the green oil for treatment, or rather to the blue oil, which is obtained from the green oil.

Each of these fractions is then subjected to a further refining operation, which consists in washing them with a small proportion of acid and soda, usually about 1 per cent., and then redistilling into fractions, which then form the commercial burning oils.

Usually the following grades of burning oils are made:—

- No. 1. Best having a gravity of 0·800, and a flash point of 100° F.
- No. 2. First quality, specific gravity, 0·810; flash point, 103° F.
- No. 3. Second quality, specific gravity, 0·815; flash point, 120° F.
- No. 4. Lighthouse oil, specific gravity, 0·820; flash point, 160° F.
- No. 5. Marine sperm oil, specific gravity, 0·830; flash point, 230° F.

After separating these fractions, there is usually left a residue of oil of about 0·840 specific gravity. This is now sold for gas making, as a fuel oil, etc.

Steam is sent into the retort to control the distillation. The oils distil off unchanged. Very little of the cracking which goes on during the distillation of American and Russian oils appears to occur with Scotch oils. The steam serves the purposes of lowering the temperature of distillation, and enables the vapour of the distilling oil to be carried out of the still. It is of great importance to fractionate the oils thoroughly. A little naphtha left in a burning oil lowers the flash point very considerably, while if any of the heavier oil is left in, it reduces the capillary power of the oil, and it burns badly. This thorough fractionation



is only effected by several distillations. The burning oils are sent out in two forms, distilled oils and treated oils. The former are purified by distillation only. These are darkest in colour and strongest in smell, but they burn the best; the wicks do not become charred so much, nor become covered with a crust. Treated oils are those which have been washed with caustic soda. They are liable to contain traces of soda, which causes them to form a crust on the wick after a time. They are clear and bright in appearance, and fairly free from odour. By careful washing of the oil with water, much of the alkali may be eliminated, in which case there is little or no crusting of the wick.

The best oils are, after being distilled and treated, subjected to a bleaching operation, which consists in exposing them in shallow tanks under a glass roofed shed to sunlight. The extent of the bleaching is not great, but it is sufficient to make the oil look paler, brighter and clearer.

A burning oil should not begin to boil much below 400° F., and leave but little residue at 572° F.

The GREEN OIL contains the lubricating oils and solid paraffin. The solid paraffin amounts to about 12 to 13 per cent. of the crude oil. It is of interest to note that, from the crude oil itself, only some 6 per cent. can be extracted, which shows that much solid paraffin must be formed by the subsequent distilling operations through which the oil passes. Probably the paraffin is present in the amorphous or uncrystallisable condition, and cannot therefore be pressed out by the usual method which is adopted for extracting paraffin; but during the distilling this amorphous paraffin is converted into crystalline paraffin. It is noticeable, too, in the same connection that the blue oil, which may be supposed to be fairly free from solid paraffin when fractionated, gives distillates which contain solid paraffin.

The green oil (or, as it is sometimes named, heavy oil and

paraffin) is run into tanks about a foot deep in large open sheds, where it is cooled. Being stirred from time to time during the operation, the solid paraffin during the cooling solidifies. From these tanks the mass is run into refrigerating tanks. These are 10 feet long by 6 feet wide. They are divided by suitable partitions into compartments. These are of two sizes—one set 6 inches wide, into which is placed the oil to be cooled, while the other set is 1 inch wide, and through these there is a constant flow of a solution of calcium chloride that has been cooled in a refrigerating machine. It is important that the rate of cooling be slow, so as to allow time for the crystals of paraffin to form properly; generally three and a half hours are allowed for the oil to cool down from 60° or 70° F. to 30° or lower, in the first refrigeration to which the oil is subjected; but in the second, or subsequent coolings, eight to nine hours are allowed. Unless the crystallisation proceeds properly, it is difficult in the subsequent pressing to press all the oil out of the scale, which then comes up softer and browner in colour than it would otherwise do. To facilitate the cooling, the sides of the tanks are scraped from time to time, and the contents of each compartment stirred up.

In some works, in place of using tanks for the purpose of air-cooling the green oil, they have long cylinders of 3 feet in diameter, in which there is a stirrer continually working. From this cylinder, when sufficiently cooled, the oil runs into the refrigerating tanks to be cooled still further.

At the Oakbank Works a somewhat different arrangement is employed for cooling and refrigerating the oil, which is allowed to take four days. The oil is run into deep but narrow tanks, built in blocks with interspaces between, these interspaces being enclosed and containing tubes through which flows a cold brine solution from a refrigerating machine. Each block is made large enough to hold a

day's production of oil. At the bottom of each cell revolves a screw worm, the shaft of which passes through stuffing-boxes at the ends of the cell and is connected with a screw worm gearing. The worm or screw in the cell is for the purpose of breaking up the solidified paraffin and getting it into a condition suitable for the filter press. This form of plant is shown in Figure 15, which represents a longitudinal section of a cell with the screw. It may be added that each block of cells is supported on girders and pillars, and under

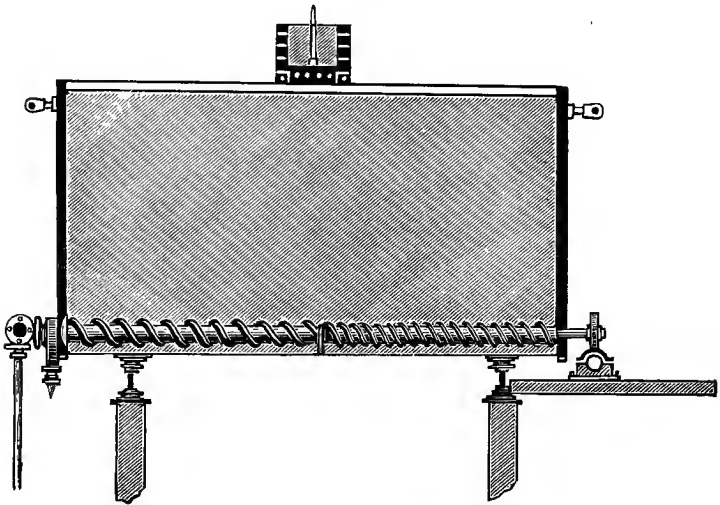


FIG. 15. Paraffin Wax Tank.

them are placed the filter presses employed to press the frozen oil. For a fuller account, a paper by Mr. George Beilby in the *Journal of the Society of Chemical Industry*, 1885, page 321, may be consulted.

After being cooled and broken up, the frozen oil is pumped through a filter press, such as is shown in Figure 16. The oil which passes through is known as blue oil, or heavy oil, while the solid paraffin that is left behind in the press is

known as "hard scale". The method of dealing with this scale will be described presently. It is important in the refining of heavy oils for lubricating that all the solid paraffin be extracted, or otherwise the specific gravity and setting point of the oil are much affected. A heavy oil of 0.863 specific gravity and setting at 42° F. will, after having 10 per cent. of paraffin removed from it, have its specific gravity raised to 0.872 and its setting point reduced to 20° F., while its viscosity will be increased. This arises from the fact that

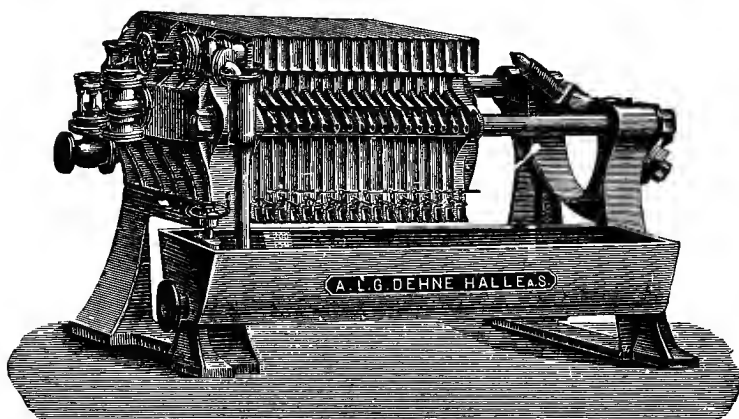


FIG. 16. Oil Filter Press.

solid paraffin when melted or in solution has a specific gravity of 0.810.

**The BLUE OIL.** The blue oil, which comes from the filter presses, usually has a specific gravity of 0.872 and a setting point of about 30° F., but it will vary according as it comes from the first cooling or the second cooling. That from the latter is higher in gravity and lower in setting point, which usually is about 20° F. In some works the blue oil is washed with acid and soda and then distilled; in others it is distilled first. This seems to be preferable to

washing it first. During the distillation it is fractionated, the following fractions being collected :—

1. 840 oil, which is used for making gas, cleaning, etc.
2. Mineral colza oil, 0·840 to 0·845 specific gravity, employed as a burning oil.
3. Lubricating oils, 0·865 specific gravity.
4.        "        "   0·875        "        "
5.        "        "   0·885        "        "
6.        "        "   0·895        "        "

The heavier oils will contain some solid paraffin. This is separated out by refrigerating them, in this case cooling down to a lower temperature than is employed with the green oil. The scale obtained is rather softer than is got from the green oil, and is known as "soft scale".

Each of the fractions may be subjected to a second distillation before it is ready to place on the market, so as to obtain as uniform a quality as possible. Further, the fractions are washed with acid and soda and then bleached by exposure to sunlight in shallow tanks under a glass roof.

As regards the special properties of the lubricating oils, these will be fully described in the chapter on Oil Testing.

The following details of the properties of the various grades of Scotch lubricating oils will be found of interest :—

"865" Oil. Light yellow in colour, with a violet-blue fluorescence.

Specific gravity at 60° F.	. . .	0·871
"        "        " 212° F.	. . .	0·816
Viscosity at 70° F.	. . .	27
"        " 100° F.	. . .	18
"        " 120° F.	. . .	16
"        " 150° F.	. . .	14
Vaporising temperature .	. . .	178° F.
Flash point . . . . .	. . .	318° F.
Fire test . . . . .	. . .	382° F.

“ 875 ” Oil. Colour, a pale yellow with violet-blue fluorescence.

Specific gravity at 60° F.	. . .	0.877
“ “ “ 212° F.	. . .	0.817
Viscosity at 70° F.	. . .	28
“ “ 100° F.	. . .	19
“ “ 120° F.	. . .	17
“ “ 150° F.	. . .	16
Vaporising point	. . .	184° F.
Flash point	. . .	330° F.
Fire test	. . .	406° F.

“ 885-90 ” Oil. Colour, yellow with violet-blue fluorescence.

Specific gravity at 60° F.	. . .	0.882
“ “ “ 212° F.	. . .	0.830
Viscosity at 70° F.	. . .	35
“ “ 100° F.	. . .	22
“ “ 120° F.	. . .	18
“ “ 150° F.	. . .	15
Vaporising temperature	. . .	190° F.
Flash point	. . .	352° F.
Fire test	. . .	440° F.

“ 890-95 ” Oil. Colour, brownish yellow with violet-blue fluorescence.

Specific gravity at 60° F.	. . .	0.892
“ “ “ 212° F.	. . .	0.840
Viscosity at 70° F.	. . .	62
“ “ 100° F.	. . .	31
“ “ 120° F.	. . .	24
“ “ 150° F.	. . .	17
Vaporising temperature	. . .	202° F.
Flash point	. . .	372° F.
Fire test	. . .	458° F.

It may however be stated here that they should not begin to distil below 600° F., or at all events but little should come over. The flash point will vary from 320° F. to 380° F., according to the gravity. Their viscosity should be good, and they ought to be as free as possible from solid paraffin, colour or smell, while their bloom or fluorescence should not be strong.

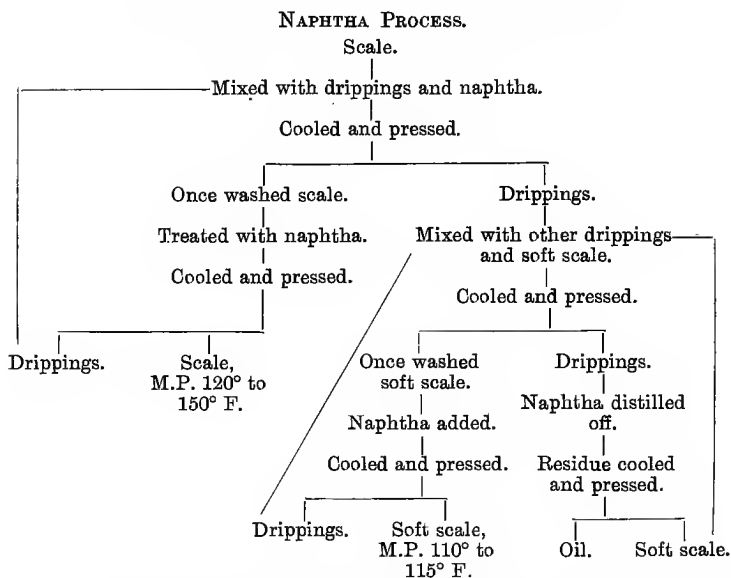
The usual grades of Scotch lubricating oils are given above. Generally, it may be said that they are of good quality, possess good flash points, which vary of course with the gravity of the oil, and have a good viscosity. They are very serviceable for lubricating all kinds of light-running machinery, shafting, etc., and are much employed for that purpose. See the chapters on Oil Testing and Lubrication.

The lubricating oils obtained from Scotch shale consist chiefly, perhaps to the extent of 80 or 90 per cent., of the higher members of the olefin series of hydrocarbons. Usually the higher members of the paraffin hydrocarbons have very little lubricating power, and the smaller the proportion of them the better is the quality of the oil produced.

The PARAFFIN SCALE is refined into paraffin wax of various melting points. One method of working is to dissolve the crude scale in naphtha, using as little as possible, then by cooling crystallising out the solid paraffin and separating it by pressure either in a filter press, or more commonly by an hydraulic press. This process is repeated several times to get the oil out of the paraffin, and so improve its melting point and colour. Sometimes the solution of the wax in naphtha is filtered through ground charcoal, or Fuller's earth, or ground spent shale, to take out the colouring matter. By steaming the solvent may be removed from the wax. Preliminary treatments with sulphuric acid and with soda have been given to the scale, but in such cases it is needful to remove by careful washing any acid or soda which may have been used.

The following diagram given by Tervet, *Journal of the Society of Chemical Industry*, 1887, p. 356, shows the general scheme of working the naphtha process of scale refining:—

## DIAGRAM OF PARAFFIN WAX REFINING.



Paraffin scale is a mixture of oily and solid paraffins of various melting points. The following table given by Tervet, *loc. cit.*, gives the melting points of various scales and waxes which have been separated into 5 per cent. fractions:—

## ANALYSES OF PARAFFIN SCALES AND WAXES.

Melting points of 5 per cent. fractions in degrees F.:—

Fraction.	Scale.	Hard Scale.	Soft Scale.	Wax 126°.	Wax 111°.	Wax 102°.
1	93	104	80	119	103	94
2	95	106	83	120	104	94
3	97	108	86	120·5	104·5	95
4	98	110	88	121	105	96
5	100	112	89	121	106	96
6	103	112·5	91	121	107	97·5
7	105·5	114	93	121·5	107·5	98
8	108	116·5	95	122	108	98·5
9	110·5	117·5	96	122·5	108·5	99
10	112·5	119	97·5	123	109	99
11	114·5	120	99	124	110·5	100
12	116	120·5	101·5	125	112	102
13	118	121	103	126	113	103·5
14	120·5	122	105	127	113·5	105
15	123	122·5	107	128	114·5	106·5
16	123·5	123·5	109·5	129	116	108
17	125	125	112	130	117	109
18	126·5	127	114	132	119	110
19	127	129	116	134	123	112·5
20	128	130	118	138	125	113



from which it will be seen that there is a wide difference in the melting points between the lowest and highest in each product. Assuming that the crude scale is simply a mechanical mixture of paraffins of various melting points, it might be considered that, by keeping the scale for some time at a low temperature, the lower melting point paraffins would be melted out, leaving the higher melting point paraffins behind. This is really the case within certain limits, and processes have been devised taking advantage of this fact; these processes being known as "sweating" processes. There are various ways of carrying out a sweating process for refining paraffin scale. Among those which have achieved any practical success are the following:—

Tervet employs an apparatus (Figure 17) which consists of two portions; the upper is a cooler consisting of a tall, but narrow, iron cistern—in this the paraffin is cooled down. It is then transferred to a sweating cell, which is formed of cloth supported by wire gauze. This cell is made large enough to hold three blocks of paraffin as they come from the coolers. These cells are placed in a room, which can be heated to any desired temperature by means of steam pipes. Owing to the heat, the oil and light paraffins in the block are melted out, and flow away through suitable pipes to a tank placed to receive them. It takes about four hours to sweat out the oil, etc. Each block of paraffin passes down into each division of the sweating cell, and as it passes down is subjected to a higher temperature, so that at each stage it gets more paraffin of low melting point taken out. In the first two cells some 35 per cent. of the original scale or wax will be taken out, while in the third cell some 10 per cent. more is taken out. The process can be regulated with great nicety. From a scale which melts at from 112° to 114° F., a wax melting at 126° F. can be readily obtained. The drippings which come out during the process are collected

and subjected to a cooling and a second sweating at rather lower temperatures, so that a wax of lower melting point is obtained ; while finally all the oil which is obtained is sent

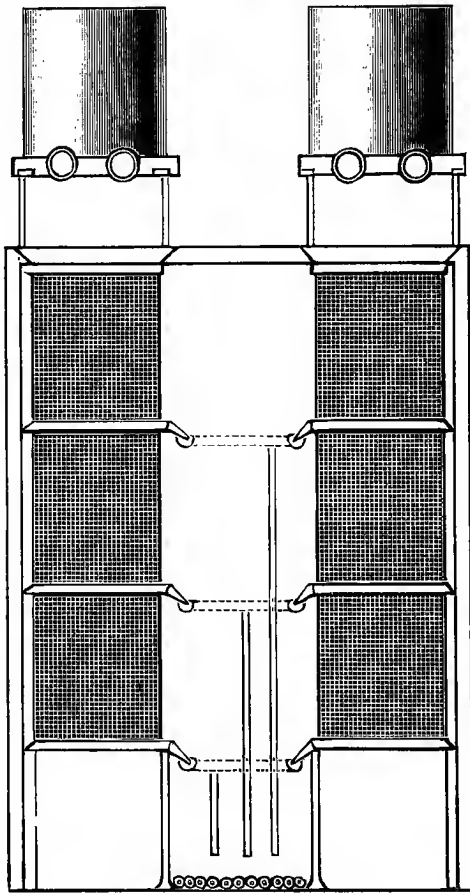


FIG. 17. Paraffin Scale Apparatus.

into the blue oil to be treated along with that material for lubricating oil.

Henderson's sweating process is carried out by placing

the solidified paraffin in metal trays in a warm chamber, heated by steam pipes to the proper degree; the melted portions or drippings run out of apertures at the end of the tray, and away out through suitable pipes. By careful regulation of the temperature of the chamber, the process may be carried on with great success, and wax of any required melting point obtained.

Usually four grades of wax are manufactured; the best has a melting point of 120° to 125° F.; the second of 115° to 120° F.; the third of 110° to 115° F.; and the fourth of 100° F.

The proportion of the various qualities of wax which are obtained is approximately as follows: 10 per cent. of 100° to 110° F., 18 per cent. of 110° to 115° F., 42 per cent. of 115° to 120° F., and 29 per cent. of 120° to 125° F. wax.

Paraffin wax consists of the higher members of the paraffin series of hydrocarbons. By a series of fractional distillations under a vacuum it is possible to separate the wax into fractions of different boiling points, and then by crystallisation from alcohol to obtain pure products. Working in this way Krafft has obtained, from a crude wax melting at 86° to 95° F., the following series of hydrocarbons, and determined their melting point, specific gravity and boiling point under a vacuum of about half an inch:—

Paraffin.	Formula.	Melting Point.	Boiling Point.	Specific Gravity at the Melting Point.
Heptadecane .	$C_{17}H_{36}$	72° F.	338° F.	0.7767
Octadecane .	$C_{18}H_{38}$	82.5° F.	359° F.	0.7768
Nonadecane .	$C_{19}H_{40}$	89.5° F.	380° F.	0.7774
Eicosane .	$C_{20}H_{42}$	98.5° F.	401° F.	0.7779
Heneicosane .	$C_{21}H_{44}$	105° F.	419° F.	0.7783
Docosane .	$C_{22}H_{46}$	112° F.	436° F.	0.7784
Tricosane .	$C_{23}H_{48}$	118° F.	453° F.	0.7785

From waxes of higher melting points, it is obvious that paraffins still higher in the series could be obtained. Gellatly

has isolated a paraffin melting at 176° F. from wax, and Gill and Meusel have obtained cerotic acid by oxidation from a wax melting at 133° F., which must therefore have contained the hydrocarbon  $C_{27}H_{56}$ .

Below will be found some statistics as to the Scotch Paraffin-Oil Industry, and the products which are obtained.

STATISTICS OF THE SCOTCH SHALE OIL INDUSTRY.  
PROGRESS OF THE INDUSTRY.

	1871. 51 works.	1879. 18 works.	1887. 13 works.	1893. ...
Shale . . . . .	Tons. 800,000	Tons. 850,000	Tons. 1,869,300	Tons. 1,947,842
Crude Oil . . . . .	Gallons. 25,000,000	Gallons. 29,000,000	Gallons. 52,876,700	Gallons. 48,696,341
Naphtha and Burn- ing Oil and Gas . . . . .	11,250,000	11,400,000	21,680,000	20,452,341
Lubricating Oil . . . . .	2,500,000	5,000,000	9,000,000	8,765,289
Paraffin, Solid . . . . .	Tons. 5800	Tons. 9200	Tons. 22,846	Tons. 19,130
Sulphate of Am- monia . . . . .	2350	4750	18,483	28,000

Capital in 1879, £1,300,000 ; in 1887, £2,000,000.

R. Irvine, *Journal of the Society of Chemical Industry*, 1894, p. 1039.

YIELDS OF PRODUCTS FROM VARIOUS CRUDE SHALE OILS.

Product.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
Naphtha . . . . .	2·00	2·00	2·00	1·75	1·55	1·80
Burning Oil . . . . .	16·75	35·00	25·80	32·75	27·85	34·10
Oil 840-850 . . . . .	3·75	4·50	4·60	4·35	4·50	5·35
Oil 865-868 . . . . .	5·50	6·00	5·80	6·15	5·40	6·20
Oil 885-890 . . . . .	17·20	12·40	15·00	10·15	14·55	3·60
Bottoms . . . . .	1·70	...	...	...	...	...
Hard Scale . . . . .	3·10	10·70	4·60	5·14	11·15	8·95
Soft Scale . . . . .	1·50	4·00	2·20	2·40	5·40	4·30
Totals . . . . .	51·50	74·60	60·00	62·69	70·40	64·30

Mills, *Destructive Distillation*, p. 42.

## PRODUCTS AND YIELDS FROM SCOTCH SHALE OIL.

Given in percentage of the Crude Oil.

Products.	Broxbourn.	Young's.
Gasoline . . . . .	...	00.25
Naphtha . . . . .	5.00	5.75
Burning Oils . . . . .	37.28	38.00
Lubricating Oils . . . . .	17.40	14.50
Wax . . . . .	12.52	11.00
Loss . . . . .	27.80	30.50

## ANALYSIS OF SHALE.

Specific gravity . . . . .	1.877
Moisture at 220° F. . . . .	2.54 per cent.
Volatile matter . . . . .	23.53 "
Fixed carbon . . . . .	12.69 "
Ash . . . . .	63.74 "

## ASH OF SHALE.

Soluble in water . . . . .	8.27 per cent.
Silica, SiO <sub>2</sub> . . . . .	53.60 "
Ferric oxide, Fe <sub>2</sub> O <sub>3</sub> . . . . .	12.23 "
Alumina, Al <sub>2</sub> O <sub>3</sub> . . . . .	22.14 "
Lime, CaO . . . . .	1.55 "
Magnesia, MgO . . . . .	trace
Sulphur . . . . .	0.94 "

The soluble portion of the ash contains 0.92 of sulphur trioxide, SO<sub>3</sub>.  
The shale contains 1.80 per cent. of sulphur, of which 1.3 remains in the ash.

The 36.22 per cent. of volatile matter and carbon contains:—

Carbon . . . . .	25.27 per cent.
Hydrogen . . . . .	3.67 "
Oxygen . . . . .	5.65 "
Nitrogen . . . . .	1.14 "
Sulphur . . . . .	0.49 "

## THE PERMANENT GAS FROM SHALE.

Composition.	No. 1.	No. 2.
Carbon Dioxide . . . . .	23.00	20.70 per cent.
Carbon Monoxide . . . . .	4.00	1.16 "
Hydrogen . . . . .	13.40	21.68 "
Olefins . . . . .	1.60	1.60 "
Marsh Gas . . . . .	19.70	8.66 "
Oxygen . . . . .	1.30	3.60 "
Nitrogen . . . . .	37.00	42.60 "

## CHAPTER IV.

### PETROLEUM.

PETROLEUM, which word is derived from two Greek words—*petros*, a rock, and *oleum*, oil; in other words, rock-oil—has long been known. It is found very widely diffused throughout the earth, in small quantities in some places, in extraordinary abundance in others. In some localities it has been known for centuries. At Zante, in the Ionian Isles, petroleum was found and was referred to by Herodotus, who flourished about 430 B.C. This deposit of petroleum is used even now locally. It is found in some quantity in Sicily; of this Plato makes mention. At Ecbatana, in Persia, petroleum is found, and of this Plato mentions that he saw it on fire. In France the deposit which occurs at Clermont and Glebian, in the department of Languedoc, has been known and used for centuries. The deposits of liquid naphtha near Baku, on the shores of the Caspian Sea, have been known for a long period, but it is only during recent years that their extraordinary abundance has been known and developed. The deposits of petroleum in Burmah have been known for centuries, and formed the first source of petroleum imported into this country. The importation was however soon stopped in consequence of the much greater supply at cheaper rates from America. In England petroleum has been found in small quantities in many localities. In 1847 a supply was found at Alfreton, in

Derbyshire, by Dr. Lyon Playfair, and was worked for a short time by James Young, of Scotch paraffin fame. At Ormskirk, in Lancashire, and in the coal mines in Northumberland, petroleum is to be found in exceedingly small deposits. Elaterite, a peculiar mineral found in an ancient lead mine in Derbyshire, is closely allied to petroleum in its composition. In Gloucestershire there are deposits of petroleum which are used locally. In California and Canada deposits of no small extent are met with. In the Argentine Republic, at Jujuy and Mandese, are lakes of asphalt, which is closely allied to petroleum; while in Trinidad is a celebrated pitch lake of some 99 acres in extent. The rise of the modern development in the use of petroleum may be ascribed to the discovery in North America by Colonel Drake, in 1858, of the rich American deposits; although prior to this the American Indians were acquainted with the use of seneca oil (crude petroleum) and used it chiefly for medicinal purposes. Since the discovery, the American petroleum industry has gone up by leaps and bounds, and now oil wells are met with over a fairly large extent of the United States—in Kentucky, New York, Michigan, Indiana, Tennessee, Colorado—the principal regions being Ohio and Pennsylvania.

The commercial petroleum oils met with in this country come almost exclusively from two sources, North America and the Caspian Sea, and it is chiefly these oils that will be dealt with in this work. Brief references may however be made to other varieties of petroleum.

#### GEOLOGY OF PETROLEUM.

Comparatively little is known concerning the conditions of formation and of the character of the rocks in which petroleum is found. In different localities even in America much is yet to be learnt concerning the age of the rocks and

the manner and formation of petroleum. So far as is known at the present time, petroleum is found in rocks of very different geological periods of time. In this respect it appears to differ materially from coal, which is found only in rocks of the carboniferous period. There are even reasons for thinking that the present deposits of petroleum have not been formed *in situ*, but have filtered from other localities into the present position of the deposits.

The general rule in Pennsylvania, New York, Ohio, Indiana and Canada is that they are stored in porous sandstones or limestones where the rocks have been gently folded into anticlinal ridges, or where, if there is a small and general dip of the strata, the dip is for a space interrupted, forming a shelf of more nearly horizontal rock, after which the strata resume their normal gentle dip.

If we trace out the underground range of these petroleum-bearing beds beyond the areas in which they are now productive, we find that they rise towards the surface and actually crop out there, but the gas and oil which they may have once contained at that out-crop have long since been lost. Like other porous rocks in such areas, they now contain water. It is the pressure of the water from the out-crop and the higher areas of the porous rock acting along and down the dip which accounts for the pressure which is met with in the gas and petroleum wells within the productive areas. When the porous bed containing gas or petroleum is tapped by a borehole, the contents are forced up by the pressure of the water from the out-crop, and the pressure depends upon the relation between the level of the out-crop and the point at which the porous bed is tapped. In fact, the condition of things somewhat resembles that so well known in the case of artesian wells.

Every richly productive gas field, at least in the eastern States and Canada, is a dome or inverted trough formed by



flexure of the rocky strata; and in every such dome or inverted trough there is a porous stratum (sandstone in Pennsylvania, and coarse-grained magnesian sandstone in Ohio and Indiana) overlain by impervious shales. These domes or arches vary in size, from a few square miles in some of the Pennsylvanian areas to 2600 square miles in the great Indiana field. Within each gas-charged dome there are found three or more substances arranged in the order of their weight: gas on the top, naphtha (if it exists on the field) and petroleum below, and finally water, which is generally salt, and which sometimes has a strong and peculiar, bitter taste. This order is invariable throughout each field, whatever its area, although in Indiana at least the oils are found more abundantly about the springing of each arch, while towards its crown gas immediately overlies brine, and the absolute altitude of the summit-level of each substance is generally uniform, whatever the depth beneath the surface. Since the volume of gas or oil accumulated in any field evidently depends on the area and height of the dome in which it is confined, and upon the porosity and thickness of rock in which it is contained, the productiveness of a given find may be definitely predicted after the structure and texture of the rocks have been ascertained.

In all productive fields the gas and oil are confined under greater or less pressure. When a gas well is closed, it is commonly found that the pressure at the well head gradually increases through a period varying from a few seconds in the largest wells to several minutes or even hours in wells of feeble flow, and after that the pressure gauge becomes stationary. This is the confined pressure, "closed pressure," or "rock pressure" of the prospector, or more properly the static pressure. When a well is open and the gas escapes freely into the air, it is found that if the stem of a mercurial or steam gauge is introduced a certain constant pressure is

indicated. This is the "open pressure" or "flow pressure" of the gas expert, and the capacity of the well may be determined from it. The static pressure varies in different fields. In Indiana it ranges from 300 to 350 lbs. per square inch, in the Findlay field it is from 450 to 500 lbs., and in the Pennsylvania field it varies from 500 to 900 lbs.

The cause of this enormous pressure is readily seen in Indiana. The Cincinnati arch (in which the gas of the great Indiana field is accumulated) is substantially a dome, about 50 miles across, rising in the centre of a stratigraphic basin fully 500 miles in average diameter. Throughout this immense basin the waters falling on the surface are in part absorbed into the rocks and conveyed towards its centre, where a strong artesian flow of water would prevail were the difference in altitude greater; and the light hydrocarbons floating upon the surface of this ground water are driven into the dome and there subjected to hydrostatic pressure equal to the weight of a column of water, whose height is the difference in altitude between the water surface within the dome and the land surface of the catchment area about the rim of the enclosing basin. Accordingly the static pressure is independent of the absolute altitude of the gas rock and of its depth beneath the surface, except in so far as these are involved in the relative altitudes of the gas rock and a catchment area perhaps scores or even hundreds of miles distant. Gas pressure and oil pressure may therefore be estimated in any given case as readily and reliably as artesian water pressure; but while the water pressure is measured approximately by the difference in altitude between catchment area and well head, that of gas is measured approximately by the difference in altitude between catchment area and gas rock, and that of oil is measured by the same difference minus the weight of a column of oil equal to the depth of the well. It follows that the static pressure of gas (as indicated on the

surface) is always greater than that of oil, particularly in deep wells. It follows also that the pressure, whether of gas or of oil, is not only constant throughout each field, but diminishes but slightly, if at all, on the tapping of the reservoir, until the supply is exhausted, and hence that pressure is no indication of either abundance or permanence of supply.

The early history of Canadian petroleum is of some interest to us, inasmuch as Dr. T. Sterry Hunt, who has studied the subject, was perhaps the first geologist who clearly understood the true geological history of American petroleum. He showed (1) that the oil was produced in or near to the beds in which it is found by the decomposition of the vegetable or animal remains; (2) that the porosity of the sandstones or limestones is sufficient to account for the great stores of petroleum which they contain; (3) that petroleum and gas mainly occur along anticlinal lines.

The comparatively simple structure of the petroleum region here described does not obtain all over the world. Often the strata in which oil occurs dip at right angles, or they have been very sharply folded and broken, the denuded edges of the petroleum-bearing bed being exposed at the surface. In such cases the yield of wells is comparatively small, there being little or no artesian pressure to force up the oil. Such regions rarely now contain much gas.

Although there is much variety of geological structure in the petroleum-bearing regions, we find that there is frequently an anticlinal arrangement of the strata, the oil coming up along the arch.

The following is a synopsis of the different shales and rocks which furnish the oil supply of North America:—

1. The black shales of the Cincinnati group afford oil which accumulates in the fissured stony limestones of the same group, and supplies the Burkesville

region of Southern Kentucky and Manitoulin Island, in Lake Huron.

2. The Marcellus shale affords most of the petroleum which accumulates in the fissured shaly limestones of the Hamilton group, and thus supplies the Ontario oil region, locally divided into the Bothwell District, the Oil Springs District and the Petrolea District. The Marcellus shale also affords a large portion of the oil which accumulates in the drift gravel of the Ontario region.
3. The Genessee shale, with perhaps some contributions from the Marcellus shale, affords oil which accumulates in cavities and fissures within itself in some of the Glasgow regions of Southern Kentucky. It affords also the oil which accumulates in the sandstones of the Portage and Chemung groups in North-western Pennsylvania and contiguous parts of Ohio. It affords also the oil which accumulates in the sandstones of the Waverly Marshall group in Central Ohio. It affords also that which accumulates in the mountain limestone of the Glasgow region of Kentucky and contiguous parts of Tennessee, and also some of that which is found in the drift gravel of the Ontario region.
4. The shaly coals of the false coal measures, aided perhaps by the Genessee and Marcellus shales, seem to afford the oil which assembles in the coal conglomerate, as worked in South-western Pennsylvania, West Virginia, Southern Ohio, and the contiguous but comparatively barren region of Paint Creek in Kentucky.

From this summary it appears that the principal supplies of petroleum east of the Rocky Mountains have been generated in four different formations, accumulated in

nine different formations, and worked in nine different districts.

The oil rocks of America belong chiefly to two periods—Devonian and Silurian—but some belong to the Cretaceous period.

In Canada the Corniferous Limestone of the Lower Devonian period is the source of the greatest proportion of the oil of Canadian origin.

In Egypt petroleum is found at Jebel Zeit, on the western border of the Red Sea, on Tertiary Strata, dipping from the range of older rocks which form the high ground of the desert.

In India petroleum is found in the Middle or Lower Tertiary rock along the flanks of the Himalayas.

In Burmah the oil is found in the Upper Tertiary Strata in soft sandy beds and covered by a blue clay situated on the top of an anticline.

The Baku District of the Caucasus is notable for its productiveness, and the rocks yielding the petroleum are found as the crown of the low anticlinal, which is probably the eastern continuation of the great Caucasian anticlinal.

The oil is found in various layers of sand, separated by clay, etc. The surface is occupied by loose sand, while the rocks below belong to the Later Tertiary period, while lower still the rocks belong to the Cretaceous and Jurassic ages.

In Roumania petroleum oil is found in the clays and sandstones of the "Paludine beds" of the Miocene age.

In Galicia petroleum is found in the rocks of the Lower Eocene age and also in smaller quantity in the Upper Cretaceous rocks. The rocks are slightly inclined, the oil being chiefly found along anticlinals.

In Hanover and other parts of Germany petroleum occurs in the Gault beds of the Jurassic period and also in rocks of the Triassic age.

## ORIGIN OF PETROLEUM.

Many theories have been put forward to account for the existence and formation of petroleum. Of these but a brief notice will be given.

Some geologists consider that it has been formed from deposits of sea-weeds, arguing from the fact that it is often found in limestone rocks, which contain the remains of ocean life. From the marine plants the petroleum has been formed much in the same way as coal has been formed from the remains of land plants.

Berthelot has promulgated a very different theory. He considers that in the interior of the earth alkali metals are present in the free condition; these acted on by carbonic acid give rise to acetylenes, which when subjected to the action of water give rise to the formation of hydrocarbons. The objection to this theory is that geologists do not find the conditions required by M. Berthelot to be present in the oil regions.

Hoefer has put forward the theory that petroleum is formed by the decomposition of animal remains. Engler has lately shown that from animal fats, free from nitrogen, petroleum can be obtained. He also points out that the absence of nitrogenous products from petroleum is an argument in favour of the theory, because the nitrogen would yield products which are insoluble and would be washed away, while the non-nitrogenous fatty portions are much more stable bodies. It may be pointed out that the gases that are found present in all oil regions contain large quantities of nitrogen with very little oxygen; this nitrogen may have been formed by the decomposition of animal matter in a peculiar manner. This theory of the origin of petroleum seems to be far the most reasonable.

## CHEMISTRY OF PETROLEUM.

Petroleum is essentially a hydrocarbon body of a most complex composition, which varies very greatly in the petroleums obtained from various localities.

While carbon and hydrogen are the essential constituents, other elements are often present; those which are most commonly present being nitrogen, oxygen and sulphur, while gold and arsenic have been found in small quantities.

The presence of sulphur beyond a small percentage is a serious disadvantage, owing to a decomposing action it has on the petroleum, giving rise to the formation of objectionable products which increase the cost of refining very greatly. The petroleums from the Lima, Ohio field, from Canada and California, are notable on account of the sulphur they contain.

Schorlemmer, in England, and Cahours, in France, have analysed American petroleum. The results obtained by Schorlemmer indicated the presence of the same hydrocarbons which are obtained by the distillation of the cannel coal, while Pelouze and Cahours showed that the distillates were all homologues of methane, or marsh gas, and belong to the series of hydrocarbons represented by the formula  $C_nH_{2n+2}$ .

Pelouze and Cahours obtained from American petroleum the following compounds:—

$C_2H_6$	.	.	.	Gas	
$C_3H_8$	.	.	.	"	
$C_4H_{10}$	.	.	.	Specific Gravity,	·600
$C_5H_{12}$	.	.	.	"	·628
$C_6H_{14}$	.	.	.	"	·669
$C_7H_{16}$	.	.	.	"	·699, Boiling Point, 92° C.
$C_8H_{18}$	.	.	.	"	·726, " " 116
$C_9H_{20}$	.	.	.	"	·741, " " 136
$C_{10}H_{22}$	.	.	.	"	·757, " " 160
$C_{11}H_{24}$	.	.	.	"	·766, " " 180
$C_{12}H_{26}$	.	.	.	"	·776, " " 200
$C_{13}H_{28}$	.	.	.	"	·792, " " 218

About the same time that these investigations were being made in France, Mr. C. M. Warren was making an exhaustive examination in America. In some respects they were confirmatory. He discovered the same compounds belonging to the  $C_nH_{2n+2}$  series. In all he succeeded in isolating fourteen different compounds in considerable quantities, sufficiently pure to allow of the separate distillation of them without any material change in the boiling point. These fourteen compounds he classifies as follows:—

FIRST SERIES.		SECOND SERIES.		THIRD SERIES.	
Formula.	Boiling Point.	Formula.	Boiling Point.	Formula.	Boiling Point.
	°C.		°C.		°C.
$C_4H_{10}$	..	$C_4H_{10}$	8·9	$C_{10}H_{20}$	174·9
$C_5H_{12}$	30·2	$C_5H_{12}$	37·0	$C_{11}H_{22}$	185·8
$C_6H_{14}$	61·3	$C_6H_{14}$	68·5	$C_{12}H_{24}$	216·2
$C_7H_{16}$	90·4	$C_7H_{16}$	98·1		
$C_8H_{18}$	119·5	$C_8H_{18}$	127·6		
$C_9H_{20}$	150·8				

It will be noticed that the compounds included in the third series do not belong to the normal paraffin series represented by the formula  $C_nH_{2n+2}$ , but to another group of hydrocarbons represented by the formula  $C_nH_{2n}$ . This is known as the ethene series or olefins.

Messrs. Warren and Storer have also examined Rangoon petroleum, in which they discovered a number of compounds of the olefin series. The following is a list of the compounds with their respective formulæ and boiling points obtained from Rangoon petroleum:—

Ruthylene, $C_{10}H_{20}$	Boiling Point, 175° C.
Margarylene, $C_{11}H_{22}$	195
Laurylene, $C_{12}H_{24}$	215
Cocinylene, $C_{13}H_{26}$	235
Naphthalene, $C_{10}H_8$	...

Also probably pelargonene ( $C_9H_{18}$ ), boiling at 155°, and



members of one or both the series of hydrocarbons (paraffins and olefins) that petroleum contains, hydrocarbons of more than one series. The American variety is almost entirely composed of the series represented by formula  $C_nH_{2n+2}$ , while the Rangoon, the Caucasian and Galician varieties contain both series; the olefins in notable quantity. As we approach the denser constituents of petroleum, the analysis becomes more difficult, and the divergency between the results of different observers is more apparent. It is now thought that paraffin, which was supposed to be a homogeneous body, is a mixture of several homologues, perhaps isomeric bodies having similar properties, but different boiling points.

Professor Henry Morton, of the Stevens Institute of Technology, has made some interesting experiments upon the "residuum" of the distillation of petroleum. Among other substances he isolated a compound to which he gave the name of "viridine". He thus speaks of it in his paper: "The crude tarry matter is well washed with benzine (petroleum naphtha), then with alcohol, and is lastly dissolved in coal tar naphtha (benzole), filtered hot and crystallised out on cooling. It is then obtained as a mass of very minute needle-like crystals of a greenish-yellow colour and pearly lustre in the mass. This I described under the name of viridine in a paper read before the American Institute in New York, and drew attention to the remarkable spectrum which its fluorescent light yielded, and which resembled in a striking manner that of anthracene, which the crystalline forms, solubility and fusing points of the two bodies were decidedly unlike." Professor Morton also expresses his belief that the substance does not "exist ready formed in the petroleum, or even in the petroleum tar, but is, like anthracene, for example, a product of destructive distillation at something like red heat".

Russian petroleum differs markedly from American petroleum in its chemical composition. It contains a few of the lower paraffin hydrocarbons, no solid paraffins, no olefins. The characteristic hydrocarbons of Russian petroleum belong to a series which are isomeric with the olefins, and having the same general formula, but differing in their constitution and properties. These have been named the naphthenes. They are allied to some extent with the hydrocarbons of coal tar. The lowest member of the series is the hexanaphthene,  $C_6H_{12}$ , which may be regarded as hexahydrobenzene. The following table gives the naphthenes that are already known:—

TABLE OF NAPHTHENES.

Formula.	Formation and Occurrence.	B.P. °C.	Spec. Gravity.
$C_6H_{12}$	Hexahydrobenzene . . . . .	69	·7539
	Russian Naphtha . . . . .	...	...
$C_7H_{14}$	Hexahydrotoluene . . . . .	97	·772
	Russian Naphtha . . . . .	95-98	·742
	Distilling Rosin . . . . .		
$C_8H_{16}$	Hexahydroxylene . . . . .	115-120	·777
	Russian Naphtha . . . . .	122-124	·7885
	Rosin Spirit . . . . .	120-123	·764
$C_9H_{18}$	Hexahydromesitylene . . . . .	135-138	...
	Hexahydropseudocumene . . . . .	135-138	·7812
	Russian Naphtha . . . . .	135-136	·7808
	Hexahydropropylbenzene . . . . .	140-142	·7811
$C_{10}H_{20}$	Dodekahydronaphthalene . . . . .	153-158	·808
	Naphtha . . . . .	160-162	·7808
	Naphtha . . . . .	168-170	·814
	From Menthene . . . . .	168-170	·797
	„ Terpene Hydrate . . . . .	168-170	·797
	„ Camphor . . . . .	167-169	·8114
	Tetrahydroterpene . . . . .	162-167	·806
$C_{11}H_{22}$	From Naphtha . . . . .	179-181	·8019
$C_{12}H_{24}$	„ „ . . . . .	197	·8120
$C_{14}H_{28}$	„ „ . . . . .	240-241	·8215
$C_{15}H_{30}$	„ „ . . . . .	246-248	·821

The characteristic features have already been described. See pp. 20 to 22.

The properties and constituents of crude petroleum vary very considerably. Some are almost colourless, limpid liquids of low specific gravities; others are heavy, dark-looking oils, while the crude ozokerite represents the extreme end of the scale by being a dark solid body. Variations are even met with in contiguous or adjacent wells—especially is this the case in the Caucasian oil fields.

Commercial petroleum oil is refined into four chief products:—

1. Naphthas, light limpid liquids, used chiefly as solvents for various purposes.
2. Burning oils, liquids varying in specific gravity from 0·805 to 0·830, used for illuminating purposes.
3. Lubricating oils of various grades.
4. Solid paraffins largely employed in candle-making. The proportion of these various groups, obtained from the crude material, varies with different varieties of petroleum.

The following details concerning the appearance and composition of various petroleums are of interest:—

#### COMPOSITION OF CRUDE PETROLEUM.

##### 1. Rangoon Oil. Specific Gravity, 0·885.

Illuminating Oil—Specific Gravity, 0·830 . . . . .	40·705 per cent.
Lubricating Oil . . . . .	40·999 „
Paraffin M.P., 60° C. . . . .	6·071 „
Tar . . . . .	4·605 „
Gas, and Loss . . . . .	7·62 „

##### 2. Enniskillen. Canada. Dark Brown.

Naphtha—Specific Gravity, 0·794 . . . . .	20 per cent.
Illuminating Oil, 0·837 . . . . .	50 „
Lubricating Oil and Paraffin . . . . .	22 „
Tar . . . . .	5 „
Carbon . . . . .	1 „
Loss . . . . .	2 „

## 3. California. Blackish. Specific Gravity, 0·927.

Illuminating Oil, 0·813	. . . . .	38 per cent.
Lubricating Oil, 0·921	. . . . .	48 „
Pitch	. . . . .	10 „
Water	. . . . .	4 „

## 4. Barbadoes Tar.

Water	. . . . .	5 per cent.
Crude Oil, 0·912	. . . . .	50 „
Gave 80 per cent. of Pale Sweet Oil, 0·908.		
Crude Oil, 0·27	. . . . .	40 „
Gave 60 per cent. Dark Oil, 0·918.		
Coke	. . . . .	5 „

5. Pennsylvania. Dark greenish, strong but not unpleasant odour.  
Specific Gravity, 0·820.

Naphtha, 0·735	. . . . .	14·7 per cent.
Burning, 0·820	. . . . .	41·0 „
Lubricating Oil	. . . . .	39·4 „
Paraffin	. . . . .	2·0 „
Coke	. . . . .	2·1 „
Loss	. . . . .	0·8 „

## 6. Canada. Dark brown, odour strong alliaceous. Specific Gravity, 0·823.

Naphtha, 0·735	. . . . .	12·5 per cent.
Burning, 0·820	. . . . .	35·8 „
Lubricating Oil	. . . . .	43·7 „
Paraffin	. . . . .	3·0 „
Coke	. . . . .	3·2 „
Loss	. . . . .	1·8 „

7. Tarentum, Pennsylvania. Dark greenish, faint pleasant odour.  
Specific Gravity, 0·820.

Naphtha, 0·723	. . . . .	4·3 per cent.
Burning, 0·820	. . . . .	44·2 „
Lubricating Oil	. . . . .	45·7 „
Paraffin	. . . . .	2·7 „
Coke	. . . . .	2·2 „
Loss	. . . . .	0·9 „

## 8. Argentine. Jujuy Lake, 88 acres. Liquid, thick, black, no disagreeable odour.

Naphtha, 150° C., 0·740	. . . . .	16 per cent.
Kerosene, 280° C., 0·830	. . . . .	34 „
Heavy Oils, 900	. . . . .	30 „
Coke	. . . . .	10 „

The following table also shows the difference in composition of various petroleum in another form. The portion distilling below 150° C. may be classed as "naphtha," that between 150° C. and 300° C. as "burning oil," that over as "lubricating oil".

COMPOSITION OF CRUDE PETROLEUMS.

District, Colour, Specific Gravity.	Commence to Boil 0° C.	Upto 150° per Cent. of Vol.	150° to 300° C.	Over 300° C.
Pennsylvania, 0·8175 . . . . .	82·0	21·0	38·25	40·75
Pennsylvania, 0·8010 . . . . .	74·0	31·5	35·0	33·5
Galicia (Sloboda), 8·235 . . . . .	90·0	26·5	47·0	26·5
Galicia (Klanczany), 189 Meters, light colour; Transparent, 0·779, containing Paraffin . . . . .	...	43·5	33·5	22·85
Galicia (Klanczany), 57 Meters, dark colour; Opaque, 0·870, no Paraffin . . . . .	...	3·4	38·6	54·5
Baku (Bibreybat), 0·889 . . . . .	91·0	23·0	38·0	39·0
Baku (Balakhan), 0·871 . . . . .	105·0	85·0	39·5	52·0
Alsace (Pechtilbrunn), 0·9075 . . . . .	135·0	3·0	50·0	47·0
Hanover (Olhheim), 0·899 . . . . .	170·0	...	32·0	68·0
Baku (Surukhanch), 0·780, amber colour . . . . .	...	50·0	45·0	...
Baku (Ilsky), 0·853 . . . . .	...	20·0	40·0	30·0

## EXTRACTION OF PETROLEUM.

Although petroleum makes its appearance here and there on the surface, yet such surface deposits are of very small extent, and do not count for much in the world's production of this valuable article. The petroleum of commerce is obtained from a kind of artesian wells, bored down to the rocky layers in which the petroleum is stored. When the petroleum well, as it is called, is to be dug, a pyramidal frame of wood of suitable height is built over the side of the well; this is termed the "derrick" (see Figure 18), and its object is to support the boring tools and the machinery for boring the well. The well is drilled by suitable boring tools, and as the boring progresses an iron tube is sent down into

the well, and it is through this iron tube that the petroleum flows out (see Figure 19). Very often the force in which petroleum is stored in the oil sands and rocks is so great as

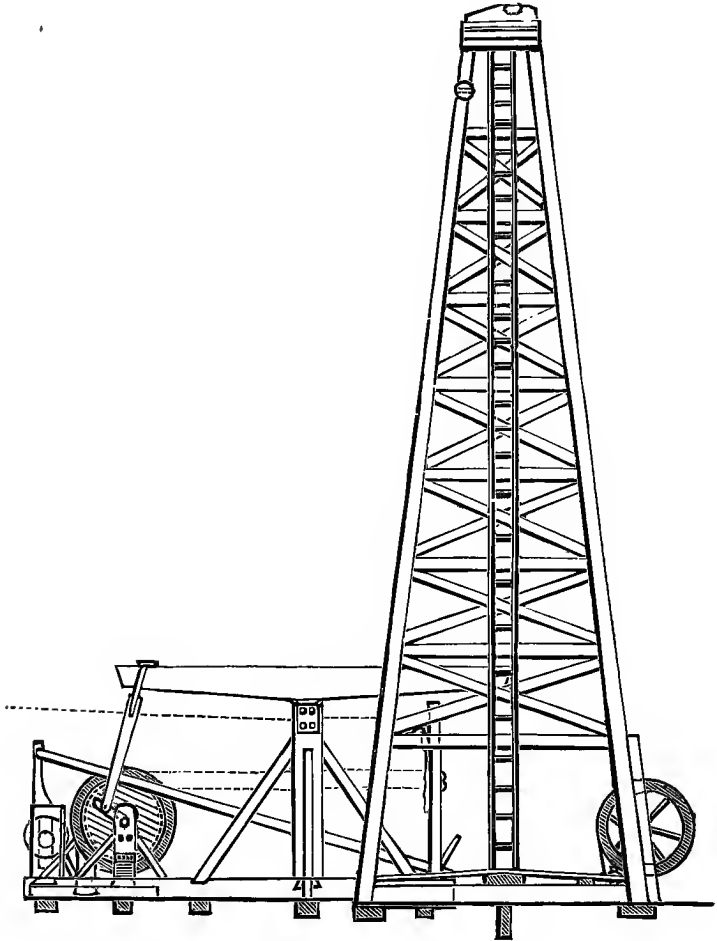


FIG. 18. Oil Well Derrick.

to cause the oil to flow out of the tube and often rise to a very considerable height above the surface of the ground.

Cases are on record both in America and Russia where the force has been so great that it has been found almost impossible to control it, and vast quantities of petroleum have been run to waste. Accompanying the flow of oil, there is always a large quantity of gas, some water and loose sand, for usually the deposits or rocks in which the oil is found are of a loose sandy nature. The crude oil as it comes from the well is run into large storage tanks, and from thence it is sent to the refiners either by means of wagons or by means of pipe lines.

#### REFINING OF PETROLEUM OILS.

Crude petroleum is a most complex substance. It contains the whole range of paraffin hydrocarbons, from liquid to solid, olefins, various basic bodies, and in some cases sulphur compounds. The process of refining aims at getting rid of the oxygen compounds, all the basic compounds and the sulphur compounds, and obtaining the paraffin and olefin hydrocarbons in the finished products. The process adopted is essentially one of distillation, accompanied by certain chemical treatments designed to rid the paraffin of the undesirable products. The principles of refining petroleum are practically identical with those of refining the Scotch shale oils. The plant used is very similar in constitution and much of it is identical, therefore it will not be necessary to describe it all in detail.

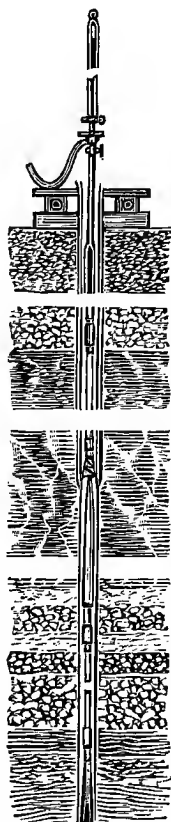


FIG. 19. Oil Well.

The exact details of the methods followed depend largely

on the character of the products desired to be obtained from the petroleum, and also upon the kind of petroleum being treated. Thus, while in the main the general principles of refining American and Russian petroleum are identical, the details are varied to suit the difference in the composition of the raw material; then again, there are some differences in the mode of treating American petroleum, according to the character of the products to be obtained from it.

#### REFINING AMERICAN PETROLEUM.

There are two methods followed in refining American petroleum, according as to whether or not it is intended to make cylinder oils and vaseline from it. To some extent the refiner is guided by the quality of his crude product. Thus some petroleums are better adapted for producing cylinder oils than others; this is notably the case with the oils from Franklin and Lima, and usually such crude oils are reserved for the manufacture of cylinder oils and vaseline.

Sometimes the refiners of crude petroleum only carry on the refining to a certain extent, leaving others to work beyond that point. This was much more common in the early days than it is now. The custom has gradually come about of the refining being conducted in very large works, capable of dealing with it in every point. Comparatively speaking, petroleum is divided into five different products:—

1. Light liquids and naphthas.
2. Kerosene and burning oils.
3. Lubricating oils.
4. Paraffin wax.
5. Coke.

The process of refining for obtaining these products is the following: The crude oil as it is received from the wells or pipe lines is run into large tanks, where it is kept warm



by means of steam pipes, and allowed to stand for some time to bring off dirt and solid matter to settle out. The oil is then run into the still and subjected to distillation. The construction of the petroleum oil still varies somewhat both in its shape, mode of setting, and method of working and size.

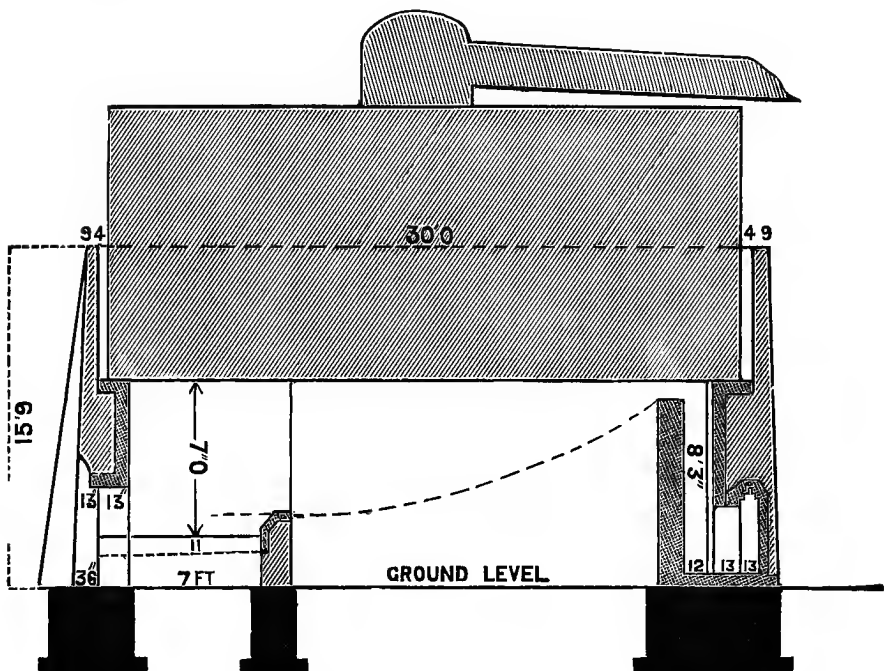


FIG. 20. Cylinder Petroleum Oil Still.

#### THE STILLS.

The two forms of stills, now almost universally used in America, are known respectively as the "cheese-box" and the "cylinder still". Advantages are claimed for both. The cylinder oil still is perhaps the most economical of fuel, and is more easily kept in repair, while the

advantages claimed for the cheese-box over the cylinder still are lighter gravity, better colour of distillates, and a larger yield of illuminating oil. The excessive cost of repairs in the brickwork and bottoms is a strong inducement to the refiner to adopt stills of the cylinder pattern, and the former are being torn down and replaced by the latter. Cylinder stills rarely exceed a capacity of 600 barrels, while some cheese-box stills have been built to contain 3500 barrels.

#### THE CYLINDER STILL.

The cylinder still is represented in Figure 20. They are frequently set in banks of two or more, there being considerable economy in thus placing them. They are 12 feet 6 inches in diameter and 30 feet in length. The capacity of this still is 600 barrels. A drum-shaped dome is usually placed in the centre of the top of the still, from which proceeds a 15-inch wrought iron pipe, connecting it with the condensing apparatus to be described further on. It will be noticed that the brickwork only extends to the centre of the still, the upper half being left wholly uncovered, or covered with a sheathing of thin sheet iron. This arrangement of the brickwork admits of the modern method of distillation being carried on, in which the process of "cracking" is an important feature.

#### THE CHEESE-BOX STILL.

This still is shown by Figures 21 and 22. It is 30 feet in diameter and 10 feet in height. It is supported by circular brickwork in which are built seventeen fireplaces, all communicating with a central flue. The bottom has a double curvature. The discharge pipe of the still enters on the side. On the inside is a swing joint suspended by a chain wound round a shaft which is operated from the outside of the still. By revolving the shaft the pipe can

either be raised or lowered to the bottom of the still. From the top of this projects three pipes, each connecting with a drum stretching across the whole diameter of the still. From this drum proceed forty 3-inch pipes leading into the condensing tanks. In some stills of both patterns, at the point where the vapours pass into the drums, a perforated steam pipe is placed. This is only employed

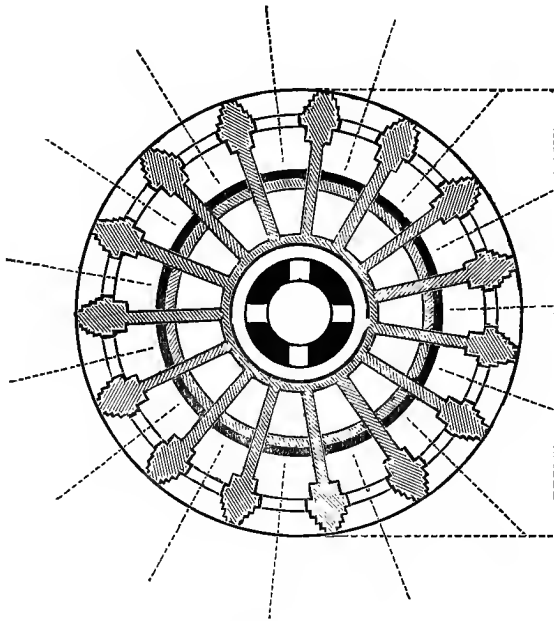


FIG. 21. Cheese-box Oil Still.

during the “cracking” process, and is thought to greatly improve the quality of the oils, both in respect to colour and gravity, although the arrangement is not to be found in many refineries.

Both forms of these stills are provided with manholes, for the double purpose of allowing the workmen to enter

and clean them, and occasionally to inspect their condition. One is placed upon the top of the still, a second near the bottom plate which allows the refuse coke to be conveniently thrown out. The covers to these are generally fastened in their places by means of screw bolts and nuts. Many of the most expert and careful refiners use pyrometers

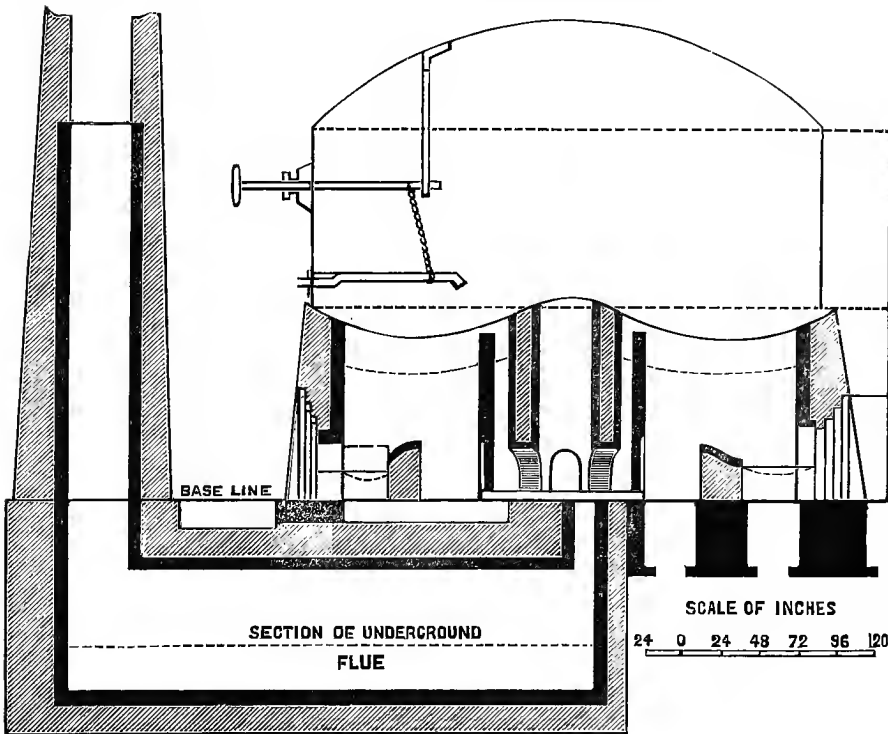


FIG. 22. Cheese-box Petroleum Oil Still.

in their stills; they are often of great assistance both to the firemen and the distiller. The large size stills are built of the best quality of boiler iron, of  $\frac{3}{8}$  or  $\frac{9}{16}$  inch thickness securely caulked. The bottoms are of steel of the same thickness. The increased expense in the use of

steel for the bottoms is more than compensated for by their greater durability and safety.

Both forms of stills are usually provided with steam pipes, both closed and perforated. The steam issuing in jets from the perforated pipe has been found to facilitate distillation by carrying over mechanically the oil vapours. Methods of continuous distillation, such as have been described on page 58 as used in the shale oil industry, have been tried for the distillation of petroleum, but have not come into much use.

The distillation of petroleum is a fractional one, but the number of the fractions which are produced varies somewhat according to the character of the products which it is desired to produce, and often to the local circumstances of the refiner, and the kind of products which he can sell best. As stated previously, in some cases the refiner only carries on the distillation to a particular point, leaving to others the further treatment.

The simplest process of oil refining consists in the fractionation into three products—1st, Naphtha; 2nd, Kerosene; 3rd, Residuum. The petroleum is distilled by fire heat, or by the combined aid of fire heat and superheated steam.

The first distillate to come over is the naphtha or benzine. From time to time the specific gravity is ascertained, and when that has reached 0.760 to 0.780 the current of the distillate is changed, and now flows into the oil tank, for what now comes over is classed as kerosene or burning oil.

The quantity of this or the extent to which the distillation is carried varies with different refiners, but the distillation is usually stopped when the specific gravity of the distillate reaches 0.84 to 0.85, although sometimes lighter distillates are collected. What remains behind in the still is

a thick, tarry mass, commonly known as "residuum". This is subsequently treated for lubricating oils.

The vapours of the distillates as they pass over from the stills are passed through a condensing arrangement, which usually takes the form of a worm pipe placed in a large tank, through which cold water is allowed to flow. This arrangement is shown in Figure 23 attached to a cylinder still. This condensing arrangement is found to work exceedingly well. For the lighter products it is sufficient to keep a flow of cold water through the tank round the worm; for the intermediate

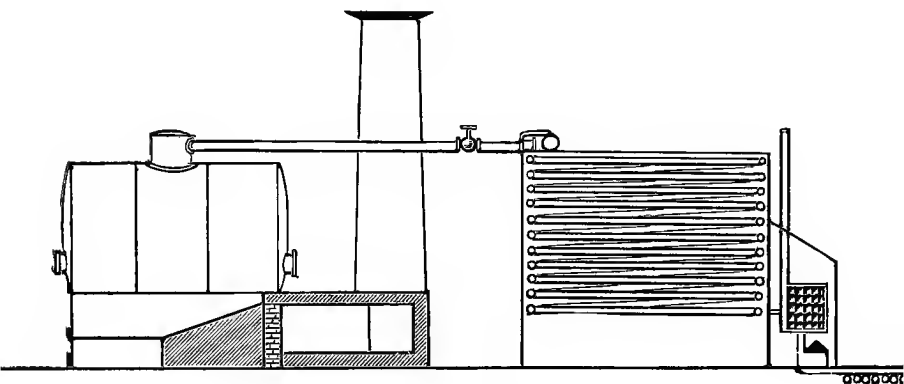


FIG. 23. Still and Condenser.

products water is not necessary, while for the heavier products obtained from the residuum, it is occasionally necessary to run hot water through the tank, with the object of preventing the solidification of the paraffin in the worm.

To separate the various fractions as they flow from the condenser an ingenious arrangement known as the monitor, shown in Figures 24 and 25, is used. As will be seen from the drawings, this consists of a circular vessel, into the bottom of which is fitted a number of exit pipes which communicate with the different tanks in which the distillates are collected.

Near the top is the pipe through which the products flow from the condenser. Inside this vessel is another which is made to revolve round a central spindle by means of a lever placed on the outside of the monitor, as shown. In this inside vessel there is a single aperture which can be brought in turn over the exit pipes in the bottom of the outer vessel. The distillates flow into the inner vessel, and thence out of the aperture into the exit pipe over which the aperture

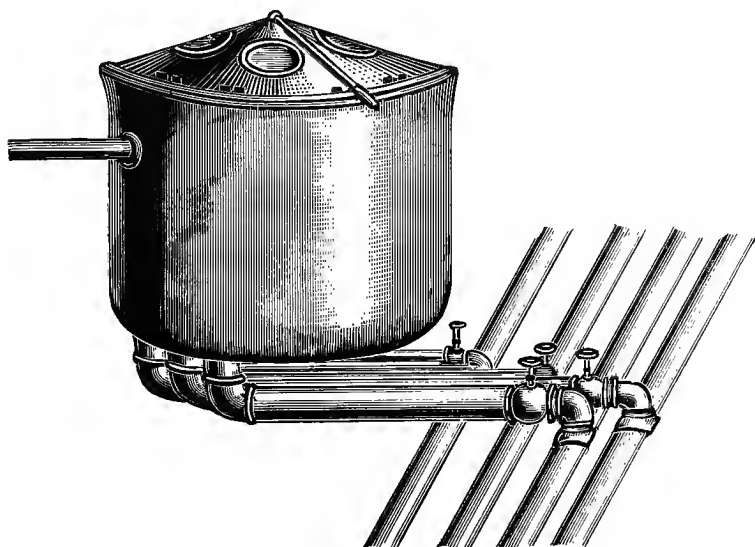


FIG. 24. Monitor or Oil Separator.

may be placed. When the direction of the flow is required to be changed, it suffices to turn the inner vessel round until the aperture is over the pipe communicating with the proper vessel. The top of the monitor is covered in with a cover containing glazed openings to see how much to turn the inner vessel to send the current of distillate in the proper direction.

Sometimes the fractionation is carried on to a much greater extent than in the above scheme, and the following distillates are obtained :—

1. Light naphtha having the specific gravity of 0·705 to 0·710. This is subsequently refined into a number of products such as cymogen, rhigolene, gasoline, light and heavy naphthas.
2. Heavy naphtha having a specific gravity from 0·705

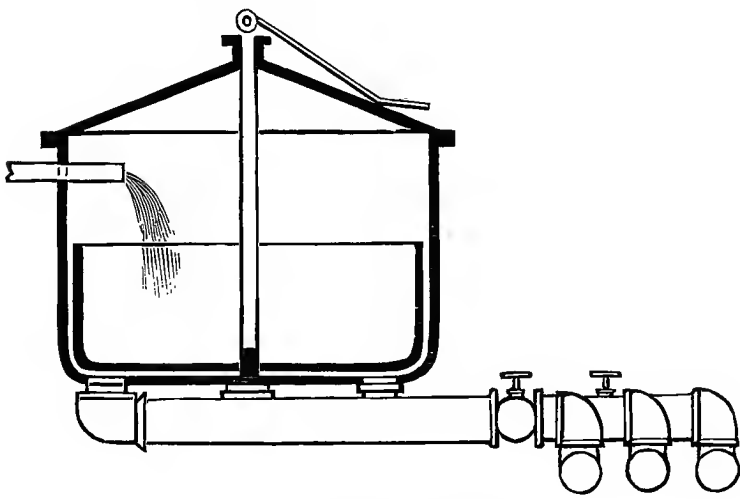


FIG. 25. Monitor or Oil Separator.

to 0·744, which also is subsequently refined into naphthas and benzines.

3. Extra heavy naphtha, the portion of the distillate ranging from 0·744 to 0·765. Used for a variety of purposes.
4. Water-white oil. Range, 0·765 to 0·795. Refined into burning oils.
5. Distillates from 0·795 to 0·825 used in preparing high flash point burning oils.



6. The distillate from 0·825, until it becomes brown, is used as gas oils.
7. Residuum.

The yield of products from the petroleum depends practically upon the composition of the crude oil, but it also depends upon the manner of distilling. If the distillation is carried on rapidly, it is found that the proportion of distillates is small, and that of the residuum large. On the other hand, if the still is built tall, and the distillation is carried on slowly, then the proportion of residuum is decreased, while that of the distillate is increased. This is due to a phenomenon which is named "cracking," and was accidentally discovered. It is due to the heat decomposing the heavier hydrocarbons, and forming in consequence lighter distillates. When the refiner desires to have a large yield of naphtha and burning oils, he usually carries on the operation in such a manner as to bring about this phenomenon of cracking. If, on the other hand, it is desired to have a large yield of lubricating oils and paraffin wax, then the cracking is avoided as far as possible. The average yields from petroleum are as follows :—

- Naphtha, 15 to 16 per cent. ;
- Burning oil, 55 per cent. ;
- Lubricating oil, 17 to 18 per cent. ;
- Paraffin wax, 2 to 3 per cent. ;

while there is some loss in the shape of uncondensed gas and coke.

The naphthas and burning oils are refined by treating them with sulphuric acid, caustic soda, and redistillation. The products which are obtained are :—

- Cymogen, a very light product, having a specific gravity of 0·590, which is used chiefly as an anæsthetic.
- Rhigolene, which has a specific gravity of 0·625, boiling

at about 65° F., is exceedingly volatile, and is used in ice-making machines and as an anæsthetic. Gasoline, having a specific gravity of 0·665, used chiefly for carburetting gas.

Naphtha or benzoline, having a specific gravity of 0·705, used as a solvent.

Benzine, having a gravity of 0·737, also used as a solvent in making varnishes and paints.

Kerosine is refined into burning oils of various grades.

#### TREATMENT OF RESIDUUM.

This is treated in various ways at different works, but the following outline will show the main lines on which it is treated. The residuum is first of all run into a large tank warmed by means of steam so as to make it fluid, then run into a still in which it is heated by fire heat alone, or in some cases fire heat accompanied by superheated steam. The whole is distilled, the distillate being condensed by means of a worm condenser, care being taken in the later periods to avoid the formation in the condensing worm of solid paraffin. There is obtained a light distillate containing solid paraffin, and a residue of coke is left behind in the still. The distilled oil is next treated with sulphuric acid and caustic soda in an upright iron vessel fitted with an agitator.

The distillate after being washed with the acid and soda is transferred to a still, again distilled, this time usually in three portions:—

1. Heavy burning oils ;
2. Light lubricating oil and paraffin ;
3. Heavy lubricating oil and paraffin ;

a residue of coke remaining in the still.

The first fraction is washed with acid and soda and refined into burning oils of various grades.

The second and third fractions are first subjected to a

refrigerating process to crystallise out the solid paraffin wax they contain, which is separated by filtration, while the liquid oil filtrate is further treated by redistillation, preceded by washing with acid and soda, and separated into lubricating oils of various grades, as follows:—

1. 875 pale oil.
2. 885 pale oil.
3. 903 to 907 pale oil.
4. 910 to 912 pale oil.

These are those usually made, but each refiner has his own special grades. These oils are commonly known in America as “paraffin oils,” in consequence of their being obtained along with paraffin with distillation.

The properties of these oils, gravities, viscosity, flash and fire tests, etc., will be described in the chapter on Oil Testing, while some details are given below.

Of late years, very considerable improvements have been made in the method of refining, a larger yield of oils of 0·885 to 0·910 gravity, with a lesser yield of oils of lower and higher gravity, being obtained. More paraffin wax is extracted, whereby an increase in the flashing point and viscosity of the oils is brought about.

#### NATURAL LUBRICATING OILS.

Some of the crude American petroleums, among which may be named those from Mecca, Ohio, Erie County, Franklin, Greensburg, Charlestown, West Virginia, Montecello, Kentucky, are of such quality that they may be used as lubricating oils with but little preparation. These oils are first allowed to settle in tanks, to free them from water and earthy impurities, when they are heated in open vessels or in stills by steam heat until they have lost all their lighter constituents and have been brought to the required degree

of gravity. These oils are dark in colour; sometimes, to reduce this somewhat, they are filtered through charcoal.

These oils are known in this country under a variety of names, of which the best known are Summer dark machinery oil, etc.

#### PETROLEUM CYLINDER OILS.

What are known as cylinder oils are a class of oils which vary in consistency from viscid liquids to soft masses of a buttery consistency, pale brown to almost black in colour, now very largely used for the lubrication of steam engine cylinders; are products obtained almost exclusively from American petroleum. In the preparation of these oils the refiner has to be careful in regard to his crude material, carefully selecting it, as it is not every variety of petroleum that yields these cylinder oils. They are produced by two methods. The first closely resembles the one described above for distilling petroleum. The crude petroleum is distilled as rapidly as possible with fire heat to get off the naphtha and the burning oil. When these have come off the distillation is aided by means of superheated steam, which carries over with it the heavier portion of the distillate, and at the same time prevents cracking, and consequently too much decomposition in the residual mass. When it is considered that the distillation is complete the residue in the still is run off, strained to free it from gritty matters, sometimes refrigerated and filtered to free it from paraffin wax, after which it is ready to be sent into commerce as "dark cylinder oil". Another method which is practised is to carry on the distillation in a vacuum whereby the light distillates are enabled to come over at a much lower temperature than

under ordinary conditions, so that there is not that decomposition of the product which sometimes occurs in the ordinary method of distilling. By a process of filtering through charcoal the colour of the product is greatly reduced in intensity. Such oils are known as "filtered" or "extra filtered" oils.

These cylinder oils vary in specific gravity from 0.888 to 0.905; their flash points are usually over 500° F., and they have a very high degree of viscosity at 100° F., but become more fluid at higher temperatures. They appear to owe their consistency to the presence of uncrystallisable members of the paraffin and olefin series of hydrocarbons. These have not been isolated. Some further details concerning these oils will be found in another section.

Similar products to the cylinder oils and practically obtained from them by repeated filtration through charcoal, are the bodies known as vaseline and petroleum jelly, largely used for medicinal purposes.

During the distillation of the cylinder oils there are obtained naphthas, burning oils, and lubricating oils. It is well known that these products are somewhat different from the lubricating oils obtained from the ordinary method of distilling residuum. They are lighter in specific gravity, ranging from 0.870 to 0.905, have a higher flashing point, and a higher viscosity. They are known in America as "neutral oils"; in England they are mostly used for the lubrication of spindles of textile spinning frames. These oils are known here under a variety of fancy names, the usual grades being 0.865, 0.872, 0.892. Details will be found in the chapter on Oil Testing.

PROPERTIES OF AMERICAN PETROLEUM.  
LUBRICATING OILS.

Grade and Brand.	Specific Gravity at		Viscosity at					Vaporising Temperature.	Flash Point.	Fire Test.	Cold Test.
	60° F.	212° F.	70° F.	100° F.	120° F.	150° F.	212° F.	Deg.	Fahr.		
<i>Pale Oils—</i>											
885 Lemon Yellow	0.883	0.833	52	27	21	16	...	210	340	452	...
903/7 Yellow . . .	0.895	0.856	102	47	32	22	...	220	386	474	...
Engine . . . . .	0.907	0.859	205	86	57	34	...	225	438	525	...
<i>Neutral Oils—</i>											
Spindle . . . . .	0.867	0.811	78	42	28	20	...	200	382	476	...
Spindle . . . . .	0.888	0.847	83	52	36	22	...	204	406	478	...
Engine . . . . .	0.899	0.858	140	61	40	25	...	210	410	486	...
Dark Cylinder . . .	0.903	...	solid	645	330	180	95	385	586	...	48
Extra Filtered . . .	0.878	0.838	solid	394	147	67	32	266	540	...	60
Dark Red Cylinder	0.895	0.849	1870	745	253	89	43	249	528	...	44
Summer Dark . . .	0.886	0.829	799	146	97	64	25	215	372	483	33
Red Oil . . . . .	0.898	0.856	197	73	40	25	17	180	420	448	...
Rope Oil . . . . .	0.870	0.822	solid	110	43	26	18	190	346	420	65

RUSSIAN PETROLEUM.

The difference in the chemical composition between American and Russian petroleums has already been pointed out.

This difference is accompanied by some difference in properties, which necessitates the crude Russian petroleum being treated by a somewhat different process to that followed with American petroleum, although the principles underlying the refining of the crude oils are much the same.

The crude Russian petroleums vary very much more in their properties than do the American petroleums. Some are light and fairly free from colour, others are heavy and of dark colour; some yield a large proportion of burning oils and little lubricating oils, while others yield a large quantity of the latter product. Russian petroleum oils are refined principally for the burning oils that can be prepared from them. Like the American product, the process of refining

consists essentially in one of distilling with chemical treatment. The chemical treatment very often cannot be carried to the same extent as is done with the American petroleum, especially the process of operating with acid, on account of the fact that the naphthenes of the Russian oil are more acted upon than the corresponding paraffins and olefins of the American oil. The distillation, again, cannot be carried on with the aid of fire heat alone, inasmuch as a consider-

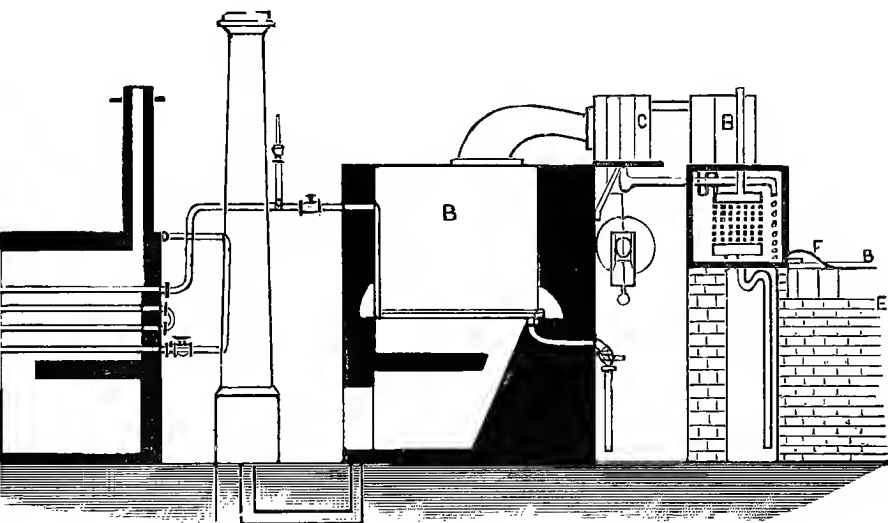


FIG. 26. Russian Petroleum Oil Refining Plant.

able amount of decomposition occurs, which is owing to the vapours being heavy and tending to flow back into the still. The distillation is carried on by the combined aid of fire heat and superheated steam, the latter acting more or less to conduct the vapours of the oil out of the still. A form of plant largely used in refining Russian petroleum is shown in Figure 26. The following is a list of the products which are obtained from Russian petroleum :—

1. Gasolines and naphthas. Specific gravity, 0·718 to 0·760; flashing under 82° F., forming about 2 per cent. of the crude oil.
2. Kerosine oils, ranging from 0·760 to 0·860 in gravity, flashing at from 82 to 136° F., forming about 38 per cent. of the crude oil, and refined into various grades of burning oils of excellent quality and high flashing points.
3. Solar oils, having a gravity of 0·860 to 0·875; flashing point, 212° F.; forming about 15 to 18 per cent. of the crude oil. This is a good illuminant when present in sufficiently large quantities, but is often mixed with astatki for use as a fuel oil and in gas making.
4. Lubricating oils, of which usually three grades are prepared.
  - (a) Spindle oils. Gravity, 0·892 to 0·896; flashing at 310 to 330° F.; forming 12 to 13 per cent. of the crude oil. These give very good results when used for lubricating textile spinning spindles provided the rate of revolution is not too high.
  - (b) Machine oils. Gravity, 0·906 to 0·908; flashing at about 340 to 350° F.; forming 27 to 28 per cent. of the product.
  - (c) Cylinder oils. Specific gravity, 0·911 to 0·912; flashing at about 390 to 400° F.; forming 4 to 5 per cent. of the crude oil. These work well for heavy shafting and engine shafts and are largely used for that purpose.
5. Tar—or, as it is known in Russia, “astatki”—the residuum left in the still, and forming 14 to 17 per cent. of the crude oil. This is used as a fuel, for caulking ships, for briquette making, grease making, and when mixed with the solar oil (see No. 3) it is used for making gas and fuel oil.



It is rather noticeable that there are no solid paraffins obtained from Russian petroleum oils, nor are there any products at all resembling the American cylinder oils. On comparing the lubricating oils from Russian petroleum and those from American petroleum and Scotch shale oil, it will be noticed that the Russian oils are somewhat higher in viscosity, which however they lose more readily when heated, while their flash points are lower than the corresponding oils of the grade from the other sources. The Russian oils do not deposit any solid paraffin at low temperatures, so that they are very suitable for use in cold places; in colour they are somewhat dark, and redder than Scotch or American oils, while their characteristic bloom is of a violet-blue tint.

The following table gives some constants relating to the various grades of Russian oils now made:—

No. 0.	
Specific Gravity at 60° F. . . . .	0.911
"    "    "    212° F. . . . .	0.862
Viscosity at 70° F. . . . .	510
"    "    100° F. . . . .	151
"    "    120° F. . . . .	87
"    "    150° F. . . . .	45
"    "    180° F. . . . .	25
Vaporising Temperature . . . . .	230° F.
Flash Point . . . . .	380° F.
Fire Test . . . . .	496° F.
No. 1.	
Specific Gravity at 60° F. . . . .	0.906
"    "    "    212° F. . . . .	0.857
Viscosity at 70° F. . . . .	320
"    "    100° F. . . . .	116
"    "    120° F. . . . .	62
"    "    150° F. . . . .	33
"    "    180° F. . . . .	22
Vaporising Temperature . . . . .	210° F.
Flash Point . . . . .	364° F.
Fire Test . . . . .	470° F.

## No. 2.

Specific Gravity at 60° F. . . . .	0.890
"    "    "    212° F. . . . .	0.846
Viscosity at 70° F. . . . .	94
"    "    100° F. . . . .	40
"    "    120° F. . . . .	28
"    "    150° F. . . . .	20
"    "    180° F. . . . .	16
Vaporising Temperature . . . . .	195° F.
Flash Point. . . . .	348° F.
Fire Test . . . . .	407° F.

## RESIDUUM.

Specific Gravity at 60° F. . . . .	0.906
"    "    "    212° F. . . . .	0.861
Viscosity at 70° F. . . . .	445
"    "    100° F. . . . .	130
"    "    120° F. . . . .	80
"    "    150° F. . . . .	38
"    "    180° F. . . . .	26

LIGHT MINERAL OILS are often "bodied up" with alkaline soaps or metallic oleates. The ordinary soaps are dehydrated by air and heat, and slowly stirred into the mineral oils heated to 180° or 200° F. in a steam-jacketed pan. If dry, the soap is completely assimilated; if still hydrated, the remaining water can be driven off by raising the temperature cautiously above 212°. Four ounces of soap will cause an ordinary 885 mineral oil to gelatinise at 60° F., and one pound of soap will convert the oil into grease.

Aluminium oleate is the thickener most used. It is made by placing 112 pounds of oleic acid into a tub containing twenty gallons of hot water, and stirring in vigorously in small quantities a lye of  $16\frac{1}{4}$  pounds, 77 per cent. caustic soda, in ten gallons of water. On boiling this mixture, sodium oleate results. Into a solution of 70 pounds of alum in 20 gallons of water this sodium oleate is now

poured, when aluminium oleate separates in greasy masses, which are skimmed off and freed of moisture by pressing. A thickening base is made by heating to 230° F. 112 pounds of oil with 28 pounds of the aluminium oleate until the latter is dissolved. The viscous product thus formed is used for bodying other oil, the mixture being facilitated by a gentle heat. One-sixteenth part by weight of aluminium oleate will give to 885 oil the body of a 910 or 915 oil. The flash point is not altered at all, and the gravity very little.

Aluminium stearate is used for the same purpose, but is more expensive and troublesome to make. It is prepared by mixing 112 pounds distilled stearine with 20 or 30 gallons of hot water and slowly adding the lye, as before described. The whole is boiled to a homogeneous mass, adding water to give the consistency of cream and make it flow freely. The sodium stearate solution thus formed is added to 20 gallons of alum solution (as above), and the aluminium stearate is skimmed off as it rises and drained on a filter, when it is ready for use.

Any residual water is driven off in the process of thickening oils, and the traces of alumina and sodium sulphate which remain in will precipitate to the bottom of the pan on standing.

While mentioning this process of thickening or bodying-up mineral oils, the author does not view the practice with favour; it gives a fictitious value to the oils, for it does not increase the lubricating power at all.

DEBLOOMING MINERAL OILS. The mineral or hydrocarbon oils possess, as has been noted in their description, a peculiar bloom or fluorescence which is very persistent. For the purpose of admixture with animal or vegetable oils, or for some uses to which they are put, this bloom is undesirable. To remove it various means have been tried. The most successful plan is to use the coal tar products, nitro-

naphthalene, binitro-benzol, and binitro-toluol, the first and last being the best. The process is simple. The oil to be debloomed is heated to 150° F. and the deblooming agent added, the amount ranging from 0.5 for oils with a slight bloom to 1.5 lb. per 100 lbs. of oil for oils with a heavy bloom. The nitro product dissolves in the oil and completely deblooms it. There is no tendency for the bloom to reappear, and the process does not appear to have any material influence on the oil.

## CHAPTER V.

### THE VEGETABLE AND ANIMAL OILS AND FATS.

#### SECTION A.

#### INTRODUCTION.

IN the bodies of all animals and in some portions of plants there is always a certain amount of oil or fatty substances, which may be extracted by suitable means. The proportion in some cases is very large, but in others is but small. Many of these oily or fatty bodies are of great value in trade, medicine, food, etc. It is only needful to mention a few—butter, tallow, olive oil, lard, sesame oil, almond oil—that are used for some such purposes as are here mentioned. The number of these bodies which are known is very great. Some are used solely for food purposes—butter, lard; others are used in medicine only—almond oil, cacao, butter; some may be employed for many purposes—olive oil, palm oil, tallow, etc. To deal with all the animal and vegetable fats and oils is beyond the scope of this book. Here will be particularly described only those which are or have been used for lubricating machinery and in the preparation of lubricating greases and compounds. In dealing generally with the chemical composition and properties of the oils and fats, others may be incidentally referred to.

## SECTION B.

## CHEMICAL COMPOSITION OF FATS AND OILS.

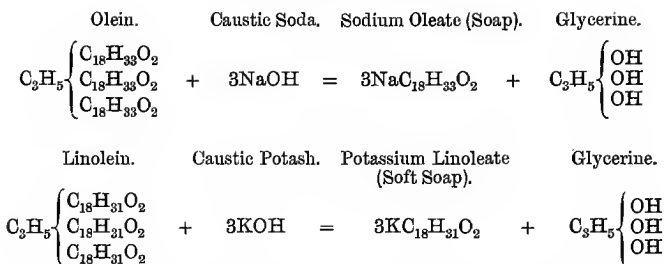
Some of the common properties of the oils and fats have been pointed out in the introductory chapter, page 2. The oils and fats now under consideration are a group of bodies which, notwithstanding their diversity of origin, are very closely related to one another. From a chemical point of view there is essentially no point of difference between an oil and a fat. In ordinary colloquial talk a fat is a body which at the ordinary temperature is a solid body like butter, while an oil is a liquid like sperm oil. But whether such a body is an oil or a fat is a question purely of temperature. Thus it happens that in cold or temperate countries like England butter and coconut oil are fats, *e.g.*, solid bodies; while in hot countries like India and Ceylon they are oils, *i.e.*, liquids. Olive oil is here an oil, in Iceland or Greenland it is a fat. At one time it was considered that there was some material difference between an oil and a fat, but it is now known that such is not the case.

The foundation of our knowledge of the chemistry of oils and fats was laid by Chevreul and Liebig in their classic researches on these bodies, and on this foundation modern chemists have built a great superstructure of knowledge, which is being added to as the years roll by. In this work English chemists have borne their share. Allen, Archbutt, Hehner, Wanklyn, Fox and others have materially helped forward this work, while continental chemists have also had a good share of the work. There is still much work remaining to be done, not only in new directions, but in confirming much of the work and researches which have been published of late years, and which if found to be correct will bring about some alterations in

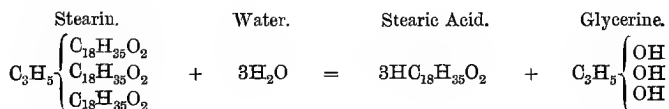
our conception of the composition of many oils. With regard to much of the statements that have been made of late of the constitution of the fatty bodies, it would be as well to be a little sceptical and await their confirmation before accepting them finally; for this reason, while the newer ideas and views will be stated in this book, the older ones will be presented at the same time.

The vegetable and animal oils and fats have been named by chemists "Glycerides," because by the processes of saponification (see farther on) they can all be made to yield glycerine. The glycerine is not present in the oils as such, but in the form of the radicle glyceryl,  $C_3H_5$ , in combination with certain acid bodies, like stearic acid and oleic acid, which are known as the fatty acids on account of their occurring in the various oils and fats. The glyceryl is the base of the oils, while the acids naturally form the acid constituent of the oils, which again belong to the great group of chemical compounds called "salts" by the chemist, and which are specially distinguished by being produced by a combination of a basic body with an acid body.

When an oil or fat is boiled with a solution of caustic soda or of caustic potash, it undergoes what is called "saponification". The stronger base soda or potash liberates glyceryl and takes its place. The compound which it then forms with the fatty acid or fatty acids of the oil or fat forms the familiar product, soap; hence the reason of the term "saponification". The glyceryl enters into combination with the hydroxyl group of the alkali and forms glycerine. This change is shown in the following equations, which show the action of caustic soda and of caustic potash on typical glycerides:—



By heating the fat in contact with superheated steam, a principle which is employed on a large scale for the manufacture of stearic acid and glycerine, the fat is decomposed, it takes up the elements of water, undergoes what is called "hydrolysis," and there is formed free fatty acids and glycerine. This reaction is shown in the following equation:—



While there is only one base present in all the fats and oils at present known—that is, glycerine, the properties of which will be found described farther on (page 123)—there are a good many acids known. In fact, with the exception of Japan wax, which is almost pure glyceryl palmitate (palmitin), most oils and fats contain at least two acids, while some, *i.e.*, butter and coconut oil, contain many more. It has been found that the acids which have so far been found in the oils and fats that have been examined, may be grouped into several families, which differ from one another in the proportion of carbon, hydrogen and oxygen they contain, and in many of their properties. The following tables give the names and chemical composition and formulæ of those acids at present known with the oils and fats in which they



have been found. In order, however, to make some of the tables complete, some acids are added which are not found in oils but are members of the same great families of acids.

I. ACIDS OF THE ACETIC SERIES. General Formula,  $C_nH_{2n}O_2$

Acid.	Formula.	Where found.
Formic . . . . .	$HCHO_2$	In ants and nettles.
Acetic . . . . .	$HC_2H_3O_2$	In various essential oils produced during the fermentation of saccharine fluids.
Propionic . . . . .	$HC_3H_5O_2$	Obtained by oxidising fusel oil.
Butyric Acid. . . . .	$HC_4H_7O_2$	The characteristic acid of butter.
Valeric Acid . . . . .	$HC_5H_9O_2$	Found in valerian root. Present in fish oils.
Caproic Acid. . . . .	$HC_6H_{11}O_2$	Obtained from butter and coconut oil.
Aenanthylic Acid . . . . .	$HC_7H_{13}O_2$	Present in castor oil.
Caprylic Acid . . . . .	$HC_8H_{15}O_2$	Found in coconut and palm nut oils, butter.
Pelargonic Acid . . . . .	$HC_9H_{17}O_2$	Oil of the pelargonium and oil of rue.
Capric Acid . . . . .	$HC_{10}H_{19}O_2$	Butter, coconut and palm nut oils.
Umbellulic Acid . . . . .	$HC_{11}H_{21}O_2$	Chaulmugra oil.
Lauric Acid . . . . .	$HC_{12}H_{23}O_2$	In laurel oil, coconut and palm nut oils.
Myristic Acid . . . . .	$HC_{14}H_{27}O_2$	In nutmeg butter, coconut oil, croton oil.
Isocetic Acid . . . . .	$HC_{15}H_{29}O_2$	Obtained from spermaceti.
Palmitic Acid . . . . .	$HC_{16}H_{31}O_2$	Palm oil, Japan wax, spermaceti.
Daturic Acid . . . . .	$HC_{17}H_{33}O_2$	
Stearic Acid . . . . .	$HC_{18}H_{35}O_2$	The solid portion of animal fats.
Arachidic Acid . . . . .	$HC_{20}H_{39}O_2$	Arachis or ground nut oil.
Behenic Acid . . . . .	$HC_{22}H_{43}O_2$	Oil of ben seed, rape oil, mustard oil.
Lignoceric Acid . . . . .	$HC_{24}H_{47}O_2$	In ground nut oil.
Hyænic Acid . . . . .	$HC_{26}H_{51}O_2$	Fat of the hyæna.
Cerotic Acid . . . . .	$HC_{27}H_{53}O_2$	Beeswax, Chinese wax, carnauba wax.
Melissic Acid . . . . .	$HC_{30}H_{59}O_2$	Beeswax.

Besides the acids here enumerated, there are reasons for suspecting the presence in some oils and fats of others more or less isomeric with some of the above, as, for instance, isobutyric acid in butter, margaric acid isomeric with daturic acid, carnaubic acid isomeric with lignoceric acid. Our knowledge of these acids is not yet sufficient to enable us to

state with sufficient definiteness the composition of all the acids described as having been found in various oils and fats. Theory, of course, points out that there is a possibility of isomeric acids existing.

The four lowest members of this series of fat acids, formic, acetic, propionic, and butyric, are soluble in water, and may be distilled without decomposition. The next few members in the series are slightly soluble in water, and may be distilled in a current of steam, while all above capryllic acid are insoluble in water, and cannot be distilled with steam. The fatty acids may therefore be divided into two divisions, one the "soluble fat acids," which are soluble in water and distillable in the presence of steam; the other the "insoluble fat acids," which are insoluble in water. The lower members, formic, acetic, propionic, and butyric acids, are liquids whose boiling points increase with an increase in the number of carbon atoms in the molecule, while the other acids are solid bodies of varying consistency whose melting point and hardness increase with increase in complexity of composition. The fatty acids are all soluble in alcohol, a property which distinguishes them from the oils and fats themselves, which are but sparingly soluble in alcohol. The solution in alcohol shows a slight acid reaction to litmus. This group of acids are saturated acids, and have no power of combination with iodine or bromine.

This group of acids have some connection with the paraffin series of hydrocarbons, inasmuch as by a series of reactions they can be prepared from them. Thus, for instance, starting with ethane,  $C_2H_6$ , it can by the action of bromine be converted into ethyl bromide,  $C_2H_5Br$ . By treating this body with caustic soda, it is converted into ethyl alcohol, ordinary alcohol,  $C_2H_5OH$ . Alcohol by treatment with oxidising agents is converted into acetic acid. In a similar manner the other hydrocarbons of the paraffin series may be con-

verted into acid bodies containing the same number of carbon atoms as the hydrocarbon.

## II. THE ACRYLIC OR OLEIC SERIES OF FATTY ACIDS.

Acid.	Formula.	Where found.
Acrylic Acid . . .	$\text{HC}_3\text{H}_3\text{O}_2$	Is obtained by oxidising acrolein.
Crotonic Acid . . .	$\text{HC}_4\text{H}_5\text{O}_2$	Is found in oil of mustard and in croton oil.
Angelica Acid . . .	$\text{HC}_5\text{H}_7\text{O}_2$	Found in Angelica root and in the resin of sumbul root.
Tiglic Acid . . . .	$\text{HC}_{13}\text{H}_{23}\text{O}_2$	In croton oil and oil of chamomile.
Moringic Acid . . .	$\text{HC}_{15}\text{H}_{27}\text{O}_2$	From oil of ben.
Cimicic Acid . . . .	$\text{HC}_{15}\text{H}_{27}\text{O}_2$	
Physetoleic Acid . .	$\text{HC}_{16}\text{H}_{29}\text{O}_2$	In sperm oil.
Hypogæic Acid . . .	$\text{HC}_{16}\text{H}_{29}\text{O}_2$	In ground nut oil or arachis oil.
Oleic Acid . . . .	$\left. \begin{array}{l} \text{HC}_{18}\text{H}_{33}\text{O}_2 \\ \dots \\ \dots \end{array} \right\}$	Is the most common of the fatty acids, the glyceride (olein) forming the liquid portion of nearly all oils.
Iso-oleic . . . .		Can be prepared from oystearic acid.
Elaidic Acid . . . .		By the action of nitrous acid on oleic acid.
Doeglic Acid . . . .	$\text{HC}_{19}\text{H}_{35}\text{O}_2$	Contained in bottlenose sperm oil.
Erucic Acid . . . .	$\text{HC}_{22}\text{H}_{41}\text{O}_2$	Rape, mustard, and grape seed oils.

Besides these acids there are some others known which can be prepared artificially, but are not known to be present in any oils or fats. This group of acids are notable on account of the large number of isomers which are known of them. Further, nearly all can, like oleic acid, be converted into an isomeric acid by the action of nitrous acid. This action is known as "elaidinising". They are unsaturated acids, each is capable of combining with chlorine, iodine, or bromine in the proportion of one molecule of the acid to two atoms of the halogen element. This property is also possessed by the oils in which they are present. The acids of the acrylic series are related to the olefin series of hydrocarbons in the same way as the acids of the acetic series are related to the paraffin hydrocarbons.

By certain reactions, as by employing hydriodic acid and phosphorus, the acids of this series can be converted into the isologous acid of the stearic series. Thus, for instance, oleic

acid, which has eighteen atoms of carbon, is converted into stearic acid, which has also eighteen atoms of carbon.

Some of the acids of this series combine directly with sulphuric acid, forming oxy-sulpho acids of the acetic series. Thus, for instance, oleic acid forms with sulphuric acid oxy-stearo-sulphuric acid,  $C_{17}H_{34}COOH.O.SO_2.OH$ , which is capable of undergoing hydrolysis and taking up the elements of water, and is transformed into sulphuric acid and oxy-stearic acid,  $C_{17}H_{34}OHCOOH$ . This reaction is of interest, as it probably takes place when soluble oil is made from olive oil by the action of sulphuric acid.

### III. THE LINOLIC SERIES OF FATTY ACIDS.

Name.	Formula.
Oleomargaric Acid . . . . .	$HC_{17}H_{29}O_2$
Linolic Acid . . . . .	$HC_{18}H_{31}O_2$
Tariric Acid . . . . .	$HC_{18}H_{31}O_2$

To these, perhaps, must be added the acid contained in millet seed oil.

These acids combine readily with iodine and bromine, taking up four atoms of the halogen per molecule of acid. They are specially characteristic of drying oils, being found in linseed, hempseed, and poppy oils. When boiled with an alkaline solution of potassium permanganate they are converted into hydroxy acids, linolic acid being converted into sativic acid. They are not elaidinised by means of nitrous acid.

### IV. THE LINOLENIC SERIES OF FATTY ACIDS.

Name.	Formula.
Linolenic Acid . . . . .	$HC_{18}H_{29}O_2$
Isolinolenic Acid . . . . .	$HC_{18}H_{29}O_2$
Jecoric Acid . . . . .	$HC_{18}H_{29}O_2$

These acids have a powerful affinity for iodine and bromine, taking up six atoms of the halogen per molecule of acid. They are found in drying oils.

### V. RICINOLEIC SERIES OF FATTY ACIDS.

Name.	Formula.
Ricinoleic Acid . . . . .	$HC_{18}H_{32}OHO_2$
Ricinisoieic Acid . . . . .	$HC_{18}H_{32}OHO_2$
Rapic Acid . . . . .	$HC_{18}H_{32}OHO_2$

These are hydroxy acids, and are allied to oleic acid. They will take up two atoms of iodine or bromine per molecule of acid. They differ from other fat acids in being insoluble in petroleum spirit, a property which also extends to their glycerides. Ricinoleic acid is characteristic of castor oil, while rapic acid is found in rape oil.

GLYCERINE, "the sweet spirit of oil," is obtained from those bodies by the process of saponification with alkalies, or by the aid of superheated steam. Chemists have assigned to this compound the name of glycerol, the termination *ol* indicating it to have alcoholic properties. Glycerine is a trihydric alcohol, and is a combination of the organic radicle glyceryl,  $C_3H_5$ , with three equivalents of hydroxyl,  $OH$ . Its chemical formula is therefore  $C_3H_5(OH)_3$ . It does not exist as such in the oils and fats; these are combinations of the radicle glyceryl with one or more fatty acids. When these undergo saponification, as shown in the equations on p. 118, the glyceryl combines with hydroxyl, and is liberated in the form of glycerine.

Glycerine possesses the following properties: It is a water-white, very viscid liquid, having a specific gravity of 1.2665 at  $15^\circ C$ . It possesses a sweet taste, but no odour. It can be obtained in the form of colourless crystals. It boils at  $290^\circ C$ . when pure. The addition of water lowers the boiling point very sensibly. It is not volatile at the ordinary temperature, but is so at the boiling point of water, and it volatilises along with water when a mixture is heated.

Glycerine is strongly hygroscopic, readily absorbing water from the air, the amount it will absorb often reaching 50 per cent. of its weight. Glycerine and water will mix in all proportions, the specific gravity varying in proportion to the degree of mixture. The following table, due to Skalweit, gives the specific gravities and refractive indices of various mixtures of glycerine and water at  $15^\circ C$ .:—

TABLE OF SPECIFIC GRAVITIES AND REFRACTIVE INDICES  
OF GLYCERINE SOLUTIONS AT 15° C.

Glycerine. Per Cent.	Specific Gravity.	Refractive Index.	Glycerine. Per Cent.	Specific Gravity.	Refractive Index.
0	1·0000	1·3330	51	1·1318	1·4010
1	1·0024	1·3342	52	1·1346	1·4024
2	1·0048	1·3354	53	1·1374	1·4039
3	1·0072	1·3366	54	1·1402	1·4054
4	1·0096	1·3378	55	1·1430	1·4069
5	1·0120	1·3390	56	1·1458	1·4084
6	1·0144	1·3402	57	1·1486	1·4099
7	1·0168	1·3414	58	1·1514	1·4104
8	1·0192	1·3426	59	1·1542	1·4129
9	1·0216	1·3439	60	1·1570	1·4144
10	1·0240	1·3452	61	1·1599	1·4160
11	1·0265	1·3464	62	1·1628	1·4175
12	1·0290	1·3477	63	1·1657	1·4190
13	1·0315	1·3490	64	1·1686	1·4205
14	1·0340	1·3503	65	1·1715	1·4220
15	1·0365	1·3516	66	1·1743	1·4235
16	1·0390	1·3529	67	1·1771	1·4250
17	1·0415	1·3542	68	1·1799	1·4265
18	1·0440	1·3555	69	1·1827	1·4280
19	1·0465	1·3568	70	1·1855	1·4295
20	1·0490	1·3581	71	1·1882	1·4309
21	1·0516	1·3594	72	1·1909	1·4324
22	1·0542	1·3607	73	1·1936	1·4339
23	1·0568	1·3620	74	1·1963	1·4354
24	1·0594	1·3633	75	1·1990	1·4369
25	1·0620	1·3647	76	1·2017	1·4384
26	1·0646	1·3660	77	1·2044	1·4399
27	1·0672	1·3674	78	1·2071	1·4414
28	1·0698	1·3687	79	1·2098	1·4429
29	1·0724	1·3701	80	1·2125	1·4444
30	1·0750	1·3715	81	1·2152	1·4460
31	1·0777	1·3729	82	1·2179	1·4475
32	1·0804	1·3743	83	1·2206	1·4490
33	1·0831	1·3757	84	1·2233	1·4505
34	1·0858	1·3771	85	1·2260	1·4520
35	1·0885	1·3785	86	1·2287	1·4535
36	1·0912	1·3799	87	1·2314	1·4550
37	1·0939	1·3813	88	1·2341	1·4565
38	1·0966	1·3827	89	1·2368	1·4580
39	1·0993	1·3840	90	1·2395	1·4595
40	1·1020	1·3854	91	1·2421	1·4610
41	1·1047	1·3868	92	1·2447	1·4625
42	1·1074	1·3882	93	1·2473	1·4640
43	1·1101	1·3896	94	1·2499	1·4655
44	1·1128	1·3910	95	1·2525	1·4670
45	1·1155	1·3924	96	1·2550	1·4684
46	1·1182	1·3938	97	1·2575	1·4698
47	1·1209	1·3952	98	1·2600	1·4712
48	1·1236	1·3966	99	1·2625	1·4728
49	1·1263	1·3981	100	1·2650	1·4742
50	1·1290	1·3996			

The refractive index of aqueous solutions of glycerine also varies with the strength, as shown in the above table, and where the quantity available is not sufficient to determine the specific gravity, this property may be made use of.

Glycerine is miscible in all proportions with alcohol ; it is insoluble in ether. It is also insoluble in carbon bisulphide, chloroform, petroleum spirit, and benzine. A mixture of two volumes of absolute alcohol and one volume of ether may be used to separate glycerine from sugars, gums, salts, etc.

Glycerine is a powerful solvent. On some bodies, *e.g.*, iodine, mercury iodide, carbolic acid, it exerts a stronger solvent action than water.

Glycerine when heated with strong sulphuric acid or with hydrogen potassium sulphate is decomposed. It suffers dehydration, and there is formed acrolein,  $C_3H_3COH$ , which has strong irritating properties. This is, perhaps, the most characteristic property of glycerine. Heated with caustic potash, potassium acetate and formate are formed. Glycerine is easily oxidised, usually with the formation of carbonic acid and water. Nitric acid forms glyceric acid ; a mixture of nitric and sulphuric acids forms nitro-glycerine, one of the most powerful explosives known. Heated with weak aqueous solutions of potassium permanganate, glycerine is converted into oxalic acid, on which fact a method for the estimation of it in waste soap, lyes, fats, etc., has been based. Full details of this test will be found described in the chapter on Oil Analysis.

In its chemical properties glycerine is a tribasic alcohol. It is capable of uniting with acids to form compounds analogous to salts. With monobasic acids like hydrochloric and acetic acids it forms three different compounds. Thus there are—

Monochlorohydrin,  $C_3H_5(OH)_2Cl$  ;  
 Dichlorohydrin,  $C_3H_5(OH)Cl_2$  ;  
 Trichlorohydrin,  $C_3H_5Cl_3$  ;

and

Monacetin,  $C_3H_5(OH)_2C_2H_3O_2$  ;  
 Diacetin,  $C_3H_5OH(C_2H_3O_2)_2$  ;  
 Triacetin,  $C_3H_5(C_2H_3O_2)_3$ .

The fatty acids are monobasic acids, and they will combine with glycerine to form three compounds. Thus from oleic acid we can get—

Monolein,  $C_3H_5(OH)_2C_{18}H_{33}O_2$  ;  
 Diolein,  $C_3H_5OH(C_{18}H_{33}O_2)_2$  ;  
 Triolein,  $C_3H_5(C_{18}H_{33}O_2)_3$ .

So far as it is at present known, only the tri-compounds of the fatty acids and glycerine are known to occur in nature.

Allen in his *Commercial Organic Analysis*, vol. ii., p. 33, gives the following figures as the proportion of glycerine which may be extracted from various oils :—

Bottlenose Sperm Oil . . . . .	3·10 per cent.
Northern Whale Oil . . . . .	11·96 „ „
Porpoise Oil . . . . .	11·09 „ „
Menhaden Oil . . . . .	11·10 „ „
Lard . . . . .	10·83 „ „
Tallow . . . . .	10·00 „ „
Butter Fat . . . . .	11·06 „ „
Olive Oil . . . . .	11·40 „ „
Rape Oil . . . . .	9·82 „ „
Sesame Oil . . . . .	9·94 „ „
Cotton Seed Oil . . . . .	9·50 „ „
Linseed Oil . . . . .	9·39 „ „
Castor Oil . . . . .	9·13 „ „
Coconut Oil . . . . .	12·11 „ „
Palm Nut Oil . . . . .	11·70 „ „
Palm Oil . . . . .	9·71 „ „
Japan Wax . . . . .	11·60 „ „
Myrtle Wax . . . . .	12·28 „ „

It will be seen that the proportion of glycerine which can be obtained from the common oils and fats is from 10 to 12 per cent.



## SECTION C.

## OCCURRENCE OF ANIMAL AND VEGETABLE OILS AND FATS.

## 1. ANIMAL FATS.

In the animal body are comparatively large deposits of fatty matter. Nearly all the internal organs are covered with a coat of fat. In and about the joints of the bones deposits of fat occur. The muscles are also separated from one another by layers of fat. In some cases these deposits of fat are of particular note. This is the case with what is known to anatomists as *omentum* of animals, a layer of fatty matter which covers the intestines, known commonly by various names—"leaf" in the case of the pig, "skin" in the case of sheep and oxen. The bodies of whales and seals are covered with a thick layer of fat known as the "blubber". The fat occurs in the animal body enclosed in small cells of animal tissue in a liquid condition, so that it does not interfere with the motions of the body. It is maintained in this liquid condition by the natural heat of the body. When the animal dies the body becomes cold, and the fatty matter sets into a solid mass, to which circumstance is due the stiffness, or *rigor mortis*, of dead bodies.

The purpose of fatty deposits in the animal kingdom is threefold. First it preserves, especially in the case of the *omentum*, the internal organs from injury and serves to lubricate them in their various motions. It serves as a store of warmth for the body; also as a store of food.

## 2. VEGETABLE OILS AND FATS.

In the vegetable kingdom oils and fats occur in a variety of ways. All seeds contain oil to a greater or less extent, some as much as 60 per cent. This oil acts as a food for the young plant until it reaches such a period of its growth that it can extract its sustenance from the

earth. The pulp of certain fruits, *e.g.*, olives, palm, contains a good deal of oil. The vegetable oils here referred to are those fixed or fatty oils, and not the essential oils, to which in many cases any particular odour or taste of the plant is due.

#### SECTION D.

##### EXTRACTION AND PURIFICATION OF ANIMAL AND VEGETABLE OILS AND FATS.

It is obvious that as the circumstances under which any particular animal or vegetable oil occurs are so varied the methods adopted for the purpose of extracting them must be varied also. A process which will work well with, say, the fat of the pig, would not suit the blubber of the whale or the oil from the olive. Space will not permit of a very extended description of all the processes which have been devised for the extraction of animal and vegetable oils and fats; but of those which are in common use a full description will be given, while other processes of only special interest will be noted in outline.

##### 1. ANIMAL FATS AND OILS.

The operation of extracting the animal oils and fats, such as tallow and lard, is generally known as "rendering". It may be carried on in various ways. The principle which underlies all the methods is that of liberating the fatty matter from the animal tissue in which it is enveloped by means of heat. This causes the fatty matter to swell, and in so doing it bursts the envelope of tissue and is then ready to flow away. We may carry this operation out by: 1st, direct heat; 2nd, boiling in water; 3rd, steam under pressure.

**RENDERING BY DIRECT HEAT.** The housewife renders her lard or suet by placing the rough fat in a tray or dish in the oven. It is scarcely possible to adopt quite so simple a system on the large scale, although one or

two plans which will be described very closely resemble the housewife's primitive method.

A simple method occasionally adopted is to heat the rough fat in a large boiler over the fire. The contents of the boiler are kept continually stirred, and when it is considered that the fat has been freed from the tissue, the fire is withdrawn and the fat drawn off into a separate receptacle. This method, though simple, is open to several objections. If great care be not taken there is a liability to char the fat or tissue, the fat thereby acquiring a discoloured appearance and a burnt odour. Not only so, but bad odours are liable to be given off during the operation which are also objectionable.

A much better plan of rendering fats by dry or direct heat is illustrated in Figure 27. A large chamber is built of such a size that a workman can conveniently enter it. In this are arranged on each side rackwork shelves placed in an inclined position towards the centre of the chamber. On the floor of the chamber are a number of steam pipes for the purpose of heating the chamber to any required degree. The fat is cut up into small fragments by means of a mincing machine, and spread in layers on metallic trays, which in turn are placed on the shelves in the chamber. At the lower end of each tray is an opening to permit of any fat running out into gutters, which are arranged for the purpose, these gutters conveying the fat into a storage tank placed in a suitable position. When all the shelves are filled with trays of fat the door of the chamber is closed, and steam sent into the pipes, whereby the chamber is heated to from 130° to 140° F. At this heat the fat melts and runs out. When it is seen that no more fat is being obtained the steam is stopped, the melted fat in the trays is allowed to run out and the residual tissue removed,

and the trays filled up for another rendering. As the residual tissue still contains some 7 or 8 per cent. of fat, it is sent to another pan for the purpose of extracting this residual fat. The advantage of this process, which has been devised by Messrs. Cook & Hall of the East London Soap Works, is that it yields a fat of very pure quality. On the other hand, it is rather more costly to work than some other processes. There is the labour

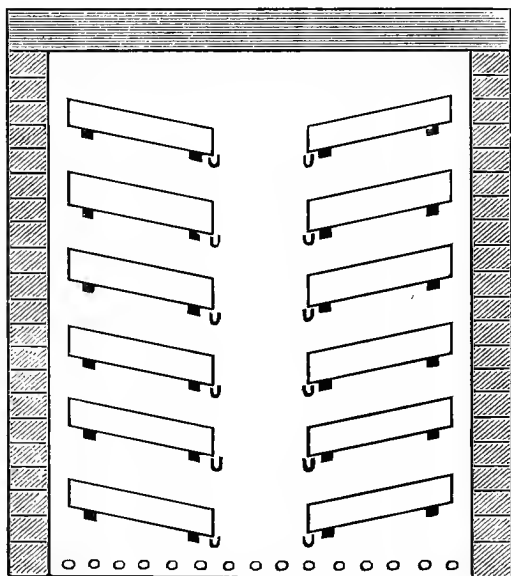


FIG. 27. Tallow Rendering Chamber.

of mincing the rough fat and filling it into the trays. The cost of heating the chamber is rather high, while the yield is not so great. A better price is however obtainable for the fat.

Messrs. Merryweather & Sons have devised a plant for the dry rendering of rough fats by superheated steam whereby the overheating of the fats is avoided. This

consists of three parts: First, a double-cased or jacketed boiler in which the fat is heated, the steam being sent into the space between the two pans; second, a superheater which is heated in a suitable furnace for superheating the steam; and, third, a steam boiler. This apparatus is very efficient in use. It is shown in Figure 28, where *h* represents the fat pan; *b*, the steam boiler; *a*, the superheater, consisting of a number of U shaped pipes in a furnace; *d* is the steam pipe; *e*, chimney. In all

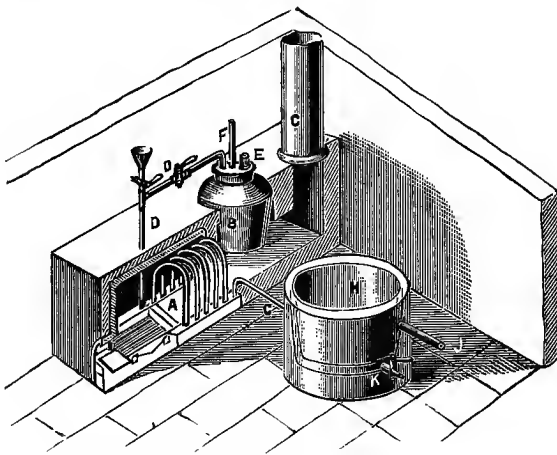


FIG. 28. Merryweather's Fat Rendering Plant.

the dry systems of rendering fats there is left behind the animal tissue, or, as it is called, the "greaves or cracklings". With the best system of rendering the fat, these always contain a certain proportion of fat which it is desirable from economical motives to recover. This may be done in several ways.

One of the most common methods of recovering the fat from greaves is by subjecting them to pressure in a press. One very convenient press for this purpose is the Boomer Screw Joint Press.

A convenient form of this press is made for fat renderers. It consists of a round table with corrugations, and provided with a lip from which the pressed fat can flow. This is supported on a strong iron casting. On the table is fitted a cask made in two halves working on a hinge, with the object of enabling it to be readily discharged. A plunger connected with the screw gear fits the cask. The Boomer press has a right- and left-handed screw, the nuts working on which are connected with the press plunger; the revolution of the screw causes the nuts to travel inwards, and thus by a knuckle joint force the plunger downwards with some force. The greaves or cracklings while still hot are placed in the cask and the press brought into action. The fat is pressed out, while the residual greaves are collected and sold for dogs' food or for manure.

RENDERING BY BOILING WATER. A very old plan of rendering tallow is to boil it in an open boiler set in a fireplace like an ordinary household washing boiler along with water. The heat of the boiling water causes the fat to expand and melt and flow from the greaves. Being lighter than water, it collects on the top of the boiling water, and should be skimmed off from time to time. The greaves fall to the bottom of the boiler. Some of the animal tissue passes into solution, however, and a little tends to get into the tallow.

This process is simple, but it has the disadvantage of leading to the production of evil odours which are objectionable, so that except for treating small lots of fat it is rarely resorted to, having been largely superseded by processes for rendering fat with steam under pressure.

Such a boiler is shown in Figure 29, which represents an improved form of construction. The bottom is double, and the space thus formed is in communication with

the outer air. By this means the temperature of the bottom of the pan never gets too high. The boiler is covered with a lid, from one portion of which a pipe conveying the steam, etc., passes into the chimney, thus carrying off all vapours and preventing nuisance.

RENDERING FATS UNDER PRESSURE BY STEAM. A very convenient form of plant for this purpose is shown in

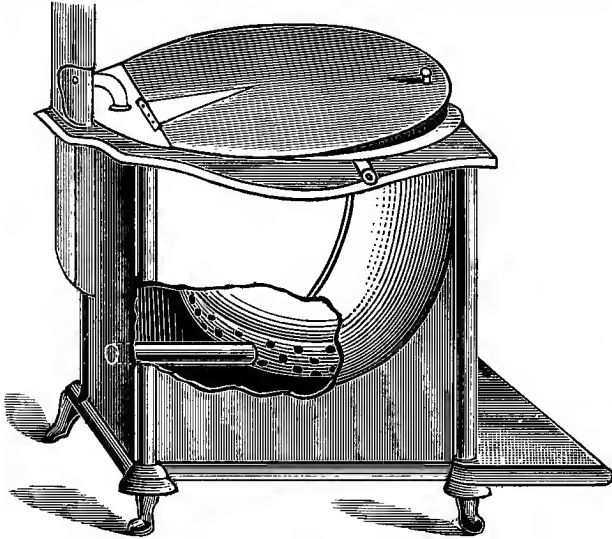


FIG. 29. Fat Boiler.

Figure 30. This consists of a steam boiler placed vertically as shown, and supported on flanges near its upper portion. The rough fat is fed in through a manhole *M*, placed on the top of the boiler, on which also is a safety valve *s*. In the bottom is placed a perforated steam coil *c*, connected with a steam-pipe and valve *v*. In the side of the boiler are placed two gauge or flow-out taps *t*, *k*, lower down is placed a large valve *R* for running off the melted tallow, while at the bottom of the boiler is another valve *T* for running off the water, etc.

This boiler is used in the following manner: The crude fat is broken up into small pieces, and fed into the boiler through the manhole, which is then closed. Water is then run in, and steam at 60 pounds pressure sent in through the steam coil; this being continued for five to six hours, the length of time being regulated according to the amount of

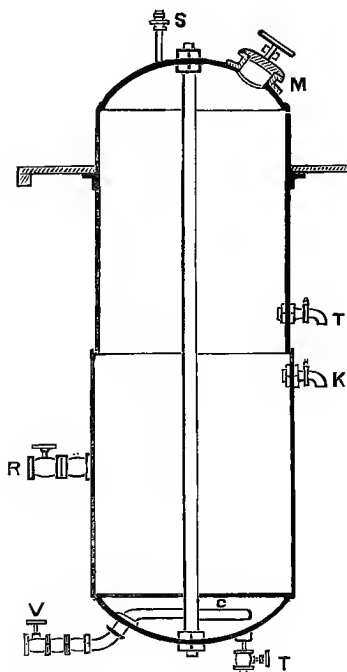


FIG. 30. Fat Boiler.

charge. When the operation is finished the steam is shut off, the contents allowed to settle, then before the fat has time to get solid it is run off from the flow-out taps. If necessary, water is run in to throw the fat up to the level of the running-off taps. Some renderers will, after turning off the steam, run off all the contents of the boiler into a tank,



and there allow the fat to settle out and solidify when it can be removed.

By using such a plant, a larger quantity of fat can be rendered in a given time ; there is less chance of objection-

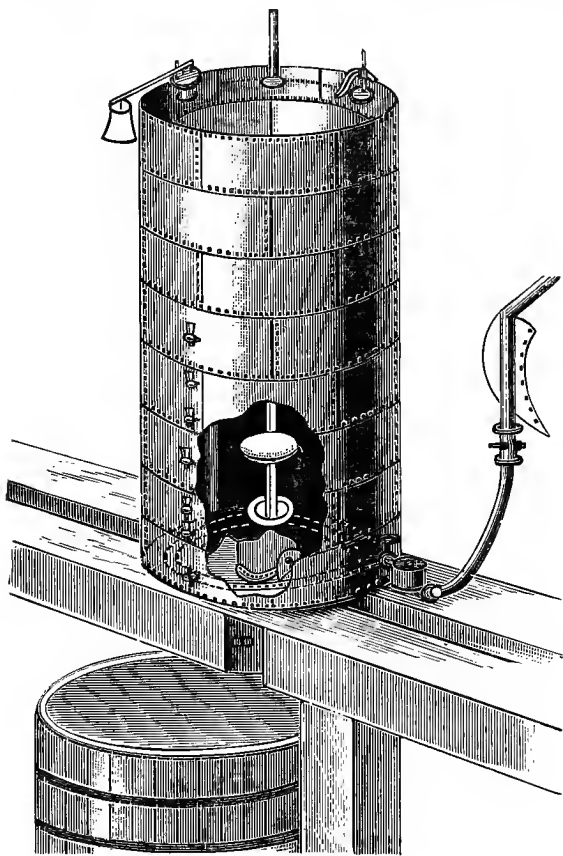


FIG. 31. Fat Boiler.

able odours arising, and the yield of rendered fat from the crude material is better.

The apparatus previously described is constructed to render the fats at the ordinary pressure, or at all events at

but slightly increased pressure. By employing boilers which are constructed to work at a higher pressure, some advantages are secured; the nitrogenous tissue is more completely gelatinised, therefore the fat is better separated from the tissue, and so a greater yield of better quality is obtained. Figure 31 represents such an apparatus, which may be built of any required size; in some cases they are constructed of a capacity of 10,000 gallons. As will be seen from the drawing, it consists of an upright boiler, fitted on the top with a safety valve, manhole for charging, and a stuffing box, through which passes the rod of the discharging orifice or valve. There is a false bottom. Between the two bottoms is a steam coil connected by valve and pipe with an ordinary steam boiler. In the bottom is a discharging orifice, which is kept closed by a plate valve worked by a rod passing through the top of the boiler. In the side of the boiler is placed a number of draw-off cocks, extending from near the bottom to about half way up, while near the top is a testing cock.

The apparatus is used in the following manner: The discharging valve is closed, and rough fat is thrown in through the manhole until the boiler is filled to within about  $2\frac{1}{2}$  feet from the top. The manhole is then closed, and steam sent in until a pressure of 45 to 60 pounds is attained. Generally a good deal of condensation of the steam occurs, and much water is formed, which collects at the bottom of the boiler. From time to time the top cock is opened. If live steam escapes the boiler is working right; if however fat comes out, then it shows that the boiler is too full, and that water must be drawn off from the lowest cock. This is done from time to time during the progress of the operation. After about twelve to fifteen hours' steaming, the steam supply is cut off, the pressure is relieved by opening the safety valve, and the contents of the boiler allowed to settle.

When well settled the water is run off, while the tallow is run into storage tanks. The aqueous liquor contains a good deal of nitrogenous matter, and it may be collected and used as manure. Working with this apparatus, there is a fairly good yield of fat or tallow from the crude material.

#### BONE TALLOW.

Bones contain a good deal of fatty matter, which it is necessary to extract before the bones can be used for other

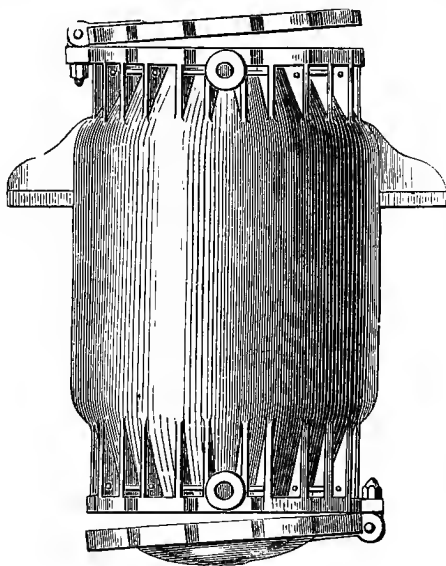


FIG. 32. Bone Boiler.

purposes. This bone fat, or, as it is commonly called, bone tallow, is very largely used in making soaps, especially soaps which are to be used for industrial purposes. The simplest plan is to adopt a boiling process in open vessels, but such a plan is open to great objection on account of the nauseous odours which are developed, therefore it is better to render bone tallow in closed vessels by steam.

Such an apparatus for the purpose is shown in Figure 32, and is constructed by Mr. W. M. Fuller of Birmingham. It consists of a boiler measuring about 6 feet by 3 feet 6 inches, fitted with hinged covers at both ends, both of which can be tightly closed by means of suitable nuts and bolts. There are also provided steam connections and draw-off cocks. A charge of about 46 cwts. of bones is put into the boiler through the upper door, which is then closed. Steam at about 50 to 60 pounds pressure is then introduced, and kept up for about forty minutes, when it is shut off; the excess steam being run into a condenser. The contents of the boiler are now allowed to settle for half an hour, when the fat is run off through a cock at the bottom of the boiler. The bones are drawn out by opening the bottom of the boiler and allowing them to drop on the floor. This boiler extracts more fatty and gelatinous matter out of the bones than does most other modes of treatment, while the bones are in a better condition for being converted into manure, being freer and therefore more friable.

In Figure 33 is shown a complete plant for the boiling and crushing of bones, as constructed by Mr. Fuller. B is a crushing mill to break up the bones prior to their being placed in the bone boilers E, E, the crushed bone being conveyed to them by means of elevators, *d*, *d* are the tanks to receive the liquor from the bones, which are passed on to the crushing and sieving mill F, where they are crushed and sieved prior to being sent out as bone meal.

Other plans of treating bones have been devised. It is quite possible that no two bone boiling establishments are arranged alike in their plant for extracting the fat and gelatine from bones.

In one works they boil the bones in pans over a fire. The pans are fitted with covers. Each pan communicates by a flue with a large iron condenser, in which all the matter

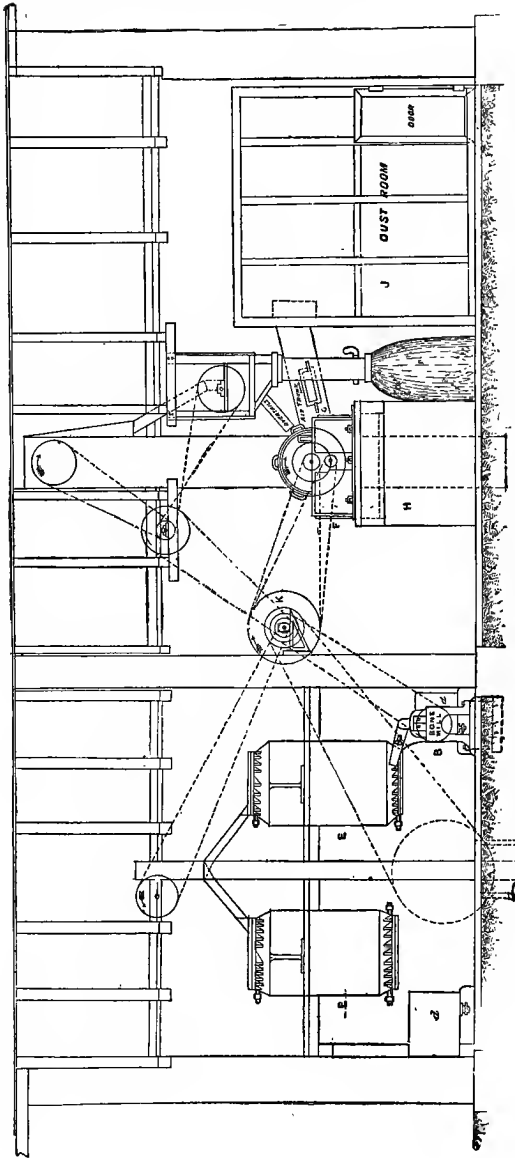


FIG. 83. Plant for Crushing and Boiling Bones

which is capable of condensing collects and flows away into suitable receptacles. Anything which is uncondensed passes into a flue and away into the atmosphere. It would be better to conduct all gases to the fireplaces to burn up all that is combustible.

In another works they use a bottle-shaped boiler, and heated by steam, both the fat and the gelatine being recovered.

The processes described above are applicable for the extraction of all kinds of animal fats, and are those usually worked. In some cases a special process may be adopted for special fats; any such will be found described under their respective fats.

## 2. VEGETABLE OILS AND FATS.

There is a greater variety in the methods of extracting oils and fats from vegetable sources than from animal sources. The method generally followed is to extract the oils by pressure, in some cases at the ordinary temperature, in others at a higher one. Some vegetable fats are extracted by a process of boiling with water, as in the case of animal fats, while the property of such bodies as carbon bisulphide, benzoline, benzol, of readily dissolving oils, is taken advantage of for obtaining vegetable oils by a solvent process.

**EXTRACTION OF VEGETABLE OILS BY PRESSURE.** The process of extracting vegetable oils by pressure is a very ancient one, and is the one commonly followed by people in a low state of civilisation. Like many other methods it has undergone many changes and developments from the earliest times to the present. It is not intended here to enter into a discussion of the changes which have taken place; attention will rather be given to the methods now in use in this country. If any reader desires to know

something of the older methods he is referred to Chambers's *Encyclopædia* or to Spon's *Dictionary of Engineering*.

There are two chief methods of pressing oils in use in this country; the oldest is generally known as the English system, the newest as the Anglo-American system.

The ENGLISH SYSTEM of oil pressing takes place in several stages as follows:—

First, Crushing.

Second, Grinding.

Third, Heating.

Fourth, Pressing.

Fifth, Refining.

The refining of the oil obtained by carrying out the first four operations is the same as in other processes of extracting oils, and will be considered later on.

*First, Crushing.* Prior to being submitted to the various operations enumerated above, the seed or other material is first subjected to a cleaning process to free it from dirt, foreign seeds, etc., which have got into it and which might interfere with the proper carrying out of the various operations or with the quality of the oil which is obtained. These cleansing processes consist essentially of winnowings and sievings through various sizes of sieves. Even with all the care that may be taken it is impossible to completely free oil seeds from all other foreign seeds, so that commercially it is doubtful whether an absolutely pure oil exists.

The crushing mills consist of an horizontal frame in which are fixed two rolls; one of these is about four feet in diameter, the other one foot in diameter. The larger roll is the driving roll, the smaller one revolving by friction against it. The two rolls are caused to press against one another with some force by means of screws and springs working against the bearings of the rolls.

The seed is fed into a hopper which delivers it between the pair of rolls, in its passage through which it becomes crushed. A mill will crush about 4 tons of seed in a working day of 10 hours. It is obvious, however, that the quantity a mill will do will vary with the kind of seed and other circumstances. It is usual to run the mill at such a speed that the large roll makes 56 revolutions

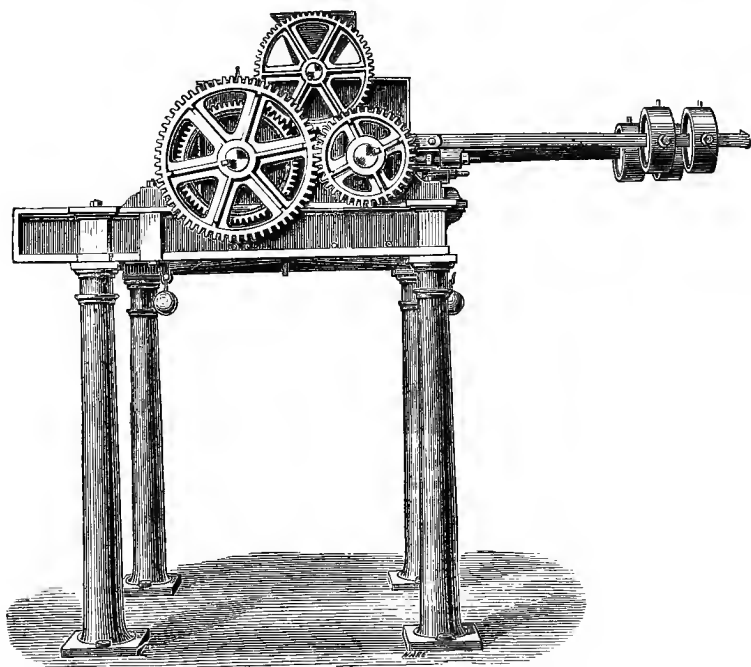


FIG. 34. Oil Seed Crushing Mill.

per minute. One of these crushing mills will keep two ordinary-sized presses at work.

Figure 34 is a drawing of such a mill as described above, made by Messrs. Rose, Downs & Thompson of the Old Foundry, Hull, to whom the author is indebted for the engraving of this and other oil machinery.



*Second, Grinding.* After being crushed the seed is thrown into the hopper of an edge runner grinding mill as shown in Figure 35, which shows the construction of such mills very well. An oil crushing mill differs from most other edge runner mills in having a shallow hopper. The usual size for the runners is 7 feet in diameter and 16 inches

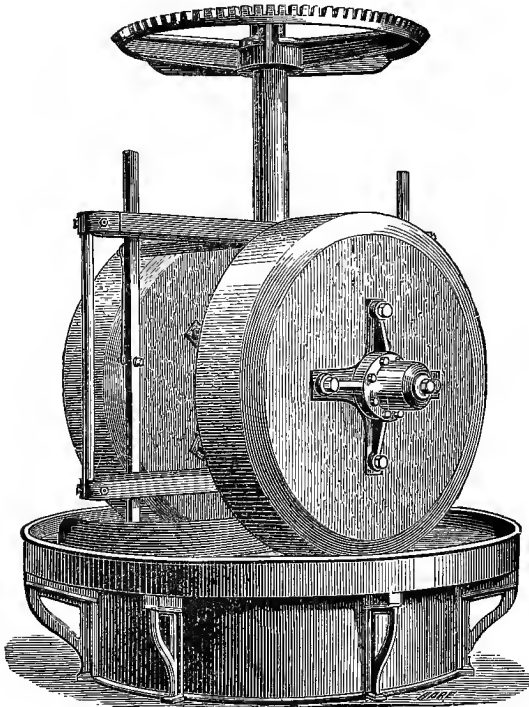


FIG. 35. Oil Seed Grinding Mill.

thick; and they will weigh 6 to 7 tons. The driving shaft makes 17 revolutions per minute. One of these edge runner mills will keep two presses at work. The seed is ground for a period of twenty to twenty-five minutes. During the operation care is taken that every part of

the seed gets efficiently ground, and it is usual to add from 2 to 3 per cent. of water during the grinding to moisten it and put the seed in the best condition for the succeeding operations.

*Third, Heating.* After being crushed and ground the seed next undergoes a heating operation. This is now

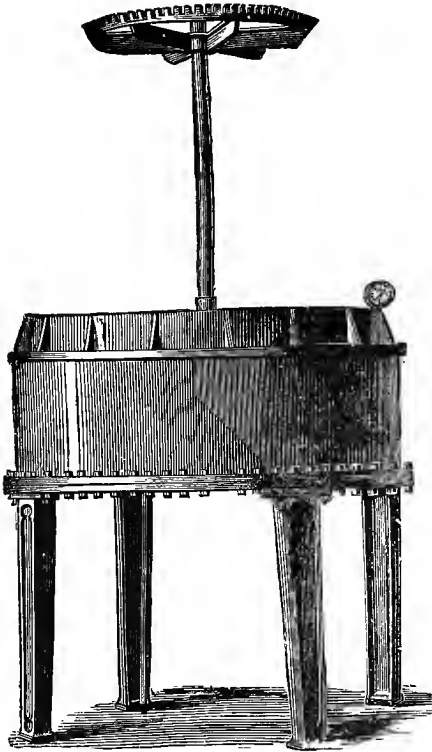


FIG. 36. Oil Seed Heating Kettle.

done in a large copper steam kettle, shown in Figure 36. This kettle varies in size according to circumstances, quantity of seed to be treated, etc. In a large oil mill the kettle will be 5 feet in diameter and 2 feet 6 inches deep. They are usually jacketed so that they can be

heated by steam up to a temperature of 160° to 170°. There is also an arrangement for sending steam into the inside of the kettle among the seed which is being treated. This is very important, as the heating in the kettle tends to dry the seed, and dry seed does not give a good yield of oil. The time of heating varies somewhat according to circumstances, but usually is about twenty to twenty-five minutes. The kettle is always fitted with an agitating apparatus to ensure that every portion of the seed is uniformly heated. One kettle of the dimensions given above will keep four presses at work capable of turning out 6 tons of cake in a day.

The previous operations are purely mechanical in their effects, being designed to get the seed into the best possible condition for yielding all the oil it contains. The heating in the kettle has a combined mechanical and chemical effect. The heating more completely breaks up the cells, and thus results in a more ready separation of the oil; while at the same time it leads to the coagulation of the albuminous and other matters present in the seed, and so prevents them from being pressed out along with the oil. It is the great object in oil pressing to obtain an oil as free as possible from extraneous vegetable matters, the presence of which in oil brings about its decomposition sooner than would otherwise be the case.

*Fourth, Pressing.* After being heated, the hot seeds are placed in strong bags made of canvas, the usual amount in each bag being 8 pounds, or sufficient seed that after pressing the oil out there remains a cake weighing 8 pounds. The bags are next enclosed in woollen covers, and are then wrapped again in what are called "hairs," which are strong cloths made of horse-hair. The cakes of seed are now placed between the plates of the press and subjected to pressure.

Beyond such primitive methods of pressing oil as have been in existence and still are in use by uncivilised people, there have been three kinds of press in use:—

First, Stamper and Wedge Press.

Second, Screw Press.

Third, Hydraulic Press.

The first and second have almost gone out of use, while now only hydraulic oil presses are made. A few words descriptive of them will however be useful.

The Stamper and Wedge Press. This old form of oil press consists of two portions. A cast iron box, long, narrow, but deep, is provided. At one end is placed a perforated iron press plate; against this is put the bag of seed; next comes another press plate, followed by a piece of wood thicker at the bottom than at the top; then comes the wedge, followed by a similar piece of wood to the foregoing. The other end is fitted up in the same way. Between the two sets is what is known as a key arrangement, consisting of three pieces, two pieces thicker at the bottom than the top, with an intermediate piece, the key, shaped like an inverted wedge. The whole of this arrangement constitutes one part. The other portion consists of two hardwood stampers, which can be made to alternately fall upon the wedges with some force, thus driving them further in and causing them to press the seed with some amount of pressure, forcing out the oil it contains. After falling on the wedge the stamper is raised up ready for another drop. When it is considered that all the oil has been extracted, a stamper is allowed to fall upon the key, which loosens the whole arrangement so that the bags of pressed seed, the oil cake as it is now called, can be withdrawn. The oil flows into a receptacle in the lower portion of the box, from whence it is transferred to a storage tank. It is

obvious of course that the production of oil in a stamper press is a limited one. About 12 cwts. per day is a fair quantity for a stamper press to turn out.

**The Screw Press.** The screw press for oil has practically gone out of use, partly because it could not be made strong enough for the work, and it was awkward to work. It usually consisted of a circular vessel in which a piston worked up and down. To the piston was attached a strong screw worked by a lever. The bags of seed were placed between the bottom of the vessel and the piston, and the screw being worked the latter was forced down, thereby pressing out the oil.

**The Hydraulic Press.** All other forms of oil presses have been virtually superseded by the hydraulic oil press, of which there are several makers. The details of the construction of the press have undergone many changes since it was first adopted for oil pressing, and the most modern form is capable of turning out more work and obtaining better products than the older ones. It will be more convenient to defer a description of the hydraulic oil press until an account of the Anglo-American system is given.

The **ANGLO-AMERICAN SYSTEM** of oil pressing was introduced into this country by Messrs. Rose, Downs & Thompson, of Hull, who have, since its introduction, greatly developed it, and made many improvements on the machinery.

The Anglo-American system requires five operations—

First, Crushing.

Second, Heating.

Third, Moulding.

Fourth, Pressing.

Fifth, Refining.

*First, Crushing.* After the seed has been cleansed, as mentioned above, p. 141, it is subjected to a crushing operation, which is given by passing it through a series of heavy

chilled iron rolls placed in a suitable frame (see Figure 37). The size and number of these rolls depend upon the quantity of seed to be dealt with. Some mills have three rolls, others four, and others five. A very common size is a mill with five rolls, each 3 ft. 6 in. long by 16 in diameter. Such a mill will pass through sufficient seed to keep a set of presses at work capable of turning out  $5\frac{1}{2}$  to 6 tons of cake in a working day.

At the top of the mill is a feeding hopper into which the

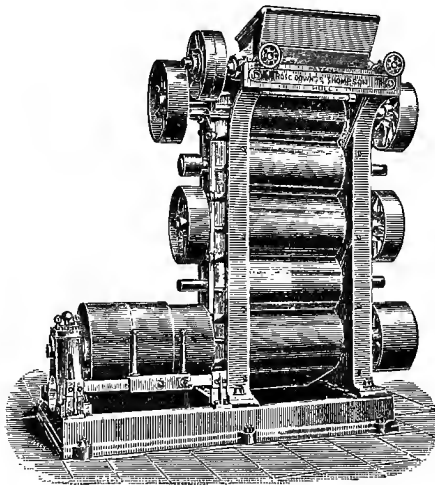


FIG. 37. Oil Seed Crushing Rolls.

seed to be crushed is placed. From this hopper it is passed between the first pair of rolls, where it receives its first crushing. By adhering to the second roll the seed is carried through between rolls Nos. 2 and 3, and successively between Nos. 3 and 4 and 4 and 5, receiving a greater crushing each time. Guides are attached to each roll to ensure that the seed is carried between each pair of rolls.

*Second, Heating.* This operation is identical with the similar operation in the English process described on p. 144.

*Third, Moulding.* After the seed has been heated in the kettle, it is sent into a moulding machine. This machine is shown in Figure 38. The moulding machine is one of the novel features of the Anglo-American system. Its object is to ensure uniformity in the size of the cakes of seed, to mould it by a gentle pressure into a level cake, so that the press is not subject to any undue strain due to inequalities in the cakes, while there is secured a greater output from the presses and a better yield of oil. The hot seed is allowed to

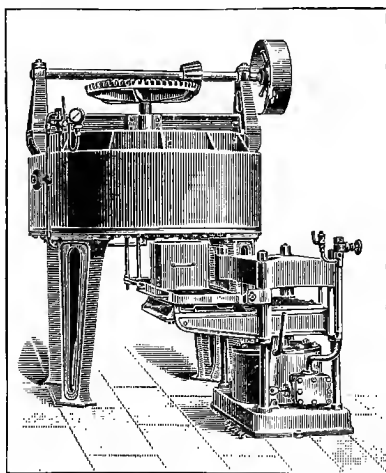


FIG. 38. Oil Cake Moulding Machine.

fall from the kettle into a measuring box, which always ensures an uniform quantity of seed being used. A tray covered with a sheet of woollen cloth is next placed on the table of the moulding machine and surrounded by a frame. Into the mould thus formed the seed is placed, and formed into a smooth cake. The tray and its contents are then pushed under the die of the moulding machine, when a cam is brought into action, and the die caused to fall upon the seed and compress it to a thickness of three inches or even

less ; the pressure being maintained for about half a minute, when the die rises and the cake of seed is removed and sent into the hydraulic press. By the use of the moulding machine a larger number of cakes can be dealt with in the press at one time. A cake of unpressed seed has a thickness of about eight inches, while the moulded seed has a thickness

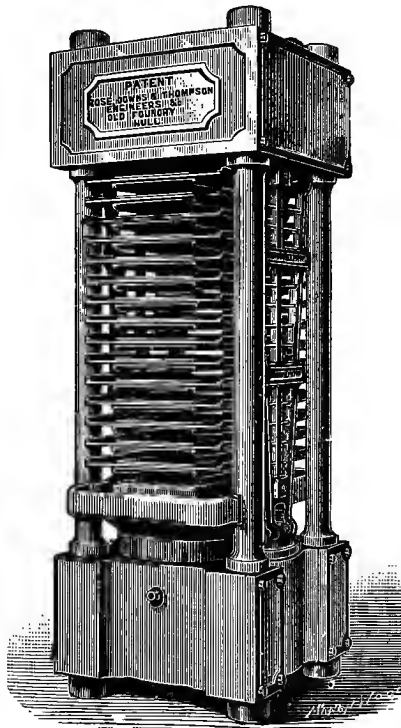


FIG. 39. Hydraulic Oil Press.

of about three inches, and so a press will take rather more than twice as much moulded seed as unmoulded seed.

*Fourth, Pressing.* This is the final operation of either the Anglo-American or the English system, although in either case the oil after it flows from the press has to undergo a



refining operation before it can be sent into the market for sale. The hydraulic press has undergone some changes in detail since it was first applied to the pressing of oil ; but it is not intended here to give any historical sketch of these changes, as space does not admit of it.

Figure 39 shows the latest form of hydraulic press for oil pressing short of the pumps which are necessary to work the press. The hydraulic press consists of a very strong cast-iron foundation, in which works a ram connection with a strong iron movable plate. This plate moves up and down between stout iron standards, which also form supports



FIG. 40. Oil Cake Moulds.

for a very strong iron casting. Between the movable bottom plate and the top are arranged a number of corrugated iron plates, which receive the cakes of seed to be pressed.

The cakes of seed as they come from the moulding machine are placed in a pair of iron covers like a book back (see Figure 40), and into the press. This does away with the hairs which were used in the old system, which are very expensive to use owing to the damage which the press does to them in breaking them. The press may be made in various sizes to suit the particular requirements of the oil

mill, from a small press capable of pressing four cakes at once to one taking twelve to fourteen cakes.

Two sets of pumps are usually supplied with each press, one to give a pressure of about 700 to 800 lbs. per square inch, while the other set will give a pressure of two tons.

At first the lower pressure is applied for about fifteen to twenty minutes, during which the great bulk of the oil will flow out. Then the higher pressure is put to complete the extraction of the oil, which will take a further five to ten minutes. The oil flows out of the sides of the seed into the corrugations on the iron plates and into channels which are provided for it to flow away to a storage or receiving tank placed about the base of the press.

Usually from two to four presses are included in one oil plant, as the other portions of the plant are quite capable of keeping more than one press at work.

It is obvious, of course, that the output from the oil press will vary very considerably owing to the varying size of the presses and also of the seed which is being pressed. For while a press can work through five charges per hour of linseed, it will only do three of rape seed and four of cotton seed in small size presses. Larger presses, working perhaps 250 to 320 lbs. of seed at one charge, will work through three to four charges in three hours. The size of the cake of seed also varies with the size of the press.

As a rule seeds are only passed once through the press, but there are a few, such as rape and gingelly seed, where the seed is crushed twice, the cake obtained in the first pressing being reground and reheated with a little additional water before being again pressed. Further, what was originally pressed in two presses is spread in the second pressing over three presses.

Some oil seeds, castor seeds, cotton seeds, arachis seeds, etc., have a very hard shell, besides being of large size. It is

necessary for the better extraction of the oil that the shell or husk be removed; this is effected by means of a machine known as a decorticator, the operation being known as "decorticating". Such a mill adapted for the treatment of castor oil seeds by hand, although they may be made to work by steam, is shown in Figure 41. These mills have a

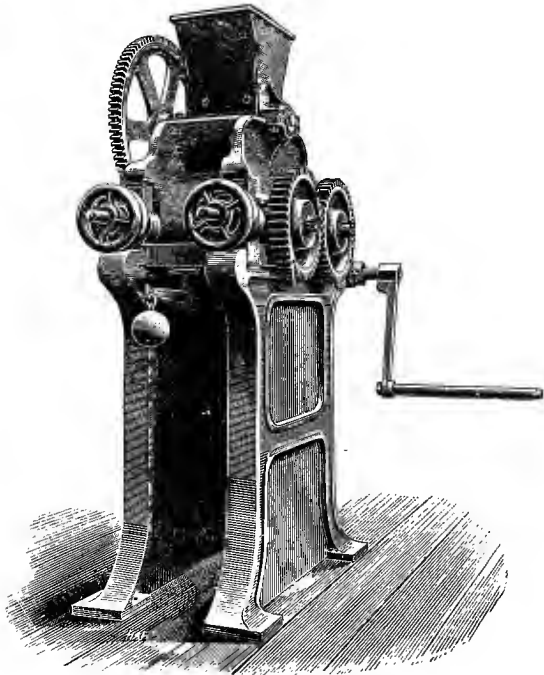


FIG. 41. Castor Seed Decorticator.

pair of revolving cylinders carrying blades fixed at a particular distance apart, this distance being dependent upon the seed which is being treated, castor oil seed requiring a different distance than arachis nuts and so on. The knives just cut the seed and allow the kernels to fall out, then by winnowing the husks may readily be separated. In Figure 42 is shown

at A castor oil seed before treatment, at B the husk, and at C the white kernel ready for the crushing mills.

There is, of course, a great difference in the quantity of oil which is yielded by different seeds. It is found preferable in the case of seeds which give but poor yields to use a smaller quantity of seed in each charge than is done with seeds which are rich in oil.

In some cases, such as castor and olive oils, the seed is subjected to two or three distinct crushings, yielding oil of several qualities. First the seed is crushed cold, when what is known as "cold drawn" or "virgin oil" is obtained; then the seed is heated and reground, when what is called

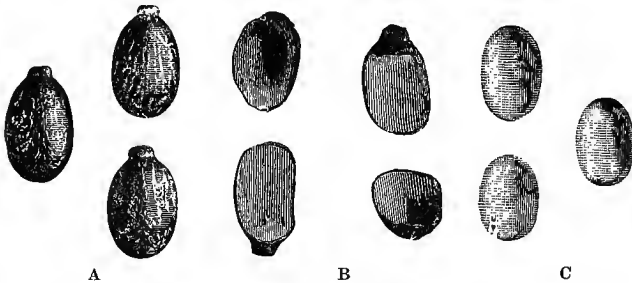


FIG. 42. Castor Seeds.

"second pressure" oil is obtained; finally, the seed is warmed with water and again pressed, when a third quality of oil is obtained.

In the case of oils which, like coconut and palm nut oils, are solid at the ordinary temperature, it is customary to heat the presses so as to make the oils fluid. In winter, too, it is desirable to work with heated presses.

The oil cake after coming from the press is passed to a paring machine, where the edges are trimmed to make the cake rather more presentable and therefore more marketable. The parings are sent back to the crushing rolls to be worked through with the next batch of seed.

## PRINCIPAL VEGETABLE OILS AND FATS.

Name of Oil, etc.	Botanical Name of Plant.	Native Country.	Percentage Yield of Oil.
Almond . . .	<i>Amygdalus communis</i>	Mediterranean Countries	48 to 50
Arachis (Earthnut, Peanut, or Groundnut)	<i>Arachis hypogaea</i>	India, Western Africa	43 to 45 (50)
Ben . . . . .	<i>Moringa oleifera</i>	India, Egypt	35 to 36
Castor . . . . .	<i>Ricinus communis</i>	East Indies	American, 46 to 49 Indian, 51 to 53
Coconut . . . .	<i>Cocos nucifera</i>	Tropical Countries	40 to 45
Colza (Rape) . .	<i>Brassica campestris</i> ; <i>B. napus</i> ; <i>B. rapa</i> ; <i>B. napobrassica</i>	Europe . . .	33 to 43
Cotton Seed . .	<i>Gossypium herbaceum</i>	Asia, Africa, America	24 to 26
Laurel Butter .	<i>Laurus nobilis</i>	South Europe	24 to 26
Linseed . . . .	<i>Linum usitatissimum</i>	Europe, Asia	38 to 40
Maize . . . . .	<i>Zea mais</i>	America . . .	6 to 10
Mustard Seed (White)	<i>Sinapis alba</i>	Europe . . .	25 to 26
Nut (Walnut) .	<i>Juglans regia</i>	Persia, Himalaya	63 to 65
Niger (Ramtil) .	<i>Guitia oleifera</i>	Abyssinia, India	40 to 45
Olive . . . . .	<i>Olea Europea</i>	Southern Europe	Pulp, 40 to 60 Kernels, 12 to 15
Palm . . . . .	<i>Elais guineensis</i>	West Africa . .	Pulp or Pericarp, 65 to 72
Poppy Seed . .	<i>E. melanococca</i> . . <i>Papaver somniferum</i>	South America . Asia Minor . .	Kernels, 45 to 50 Blue, 48 to 50 White, 41 to 45
Sesame (Gingelly)	<i>Sesamum indicum</i>	India, Levant, Antilles, etc.	50 to 57
Sunflower Seed .	<i>Helianthus annuus</i>	Mexico, Peru .	21 to 22

REFINING AND CLARIFYING OILS. The oils as they come from the oil press are usually cloudy in appearance, contain much colouring matter, moisture and extraneous vegetable tissue, from which it is desirable they should be freed.

The oil as it comes from the press should be run into large tanks, which are kept at about 100° F., in which it is allowed to remain for some time. The water and solid vegetable matter settles down, leaving the oil bright and clear. This process is however a slow one, and the oil

presser often does not care to keep his oils so long, and therefore must adopt a quicker process.

Sometimes oils are clarified by adding to them from 5 to 10 per cent. of their weight of fuller's earth, heating the mixture to about 150° F., and maintaining it at that heat for half an hour to an hour, stirring well all the time. Then the oil is allowed to stand for about twenty-four to thirty hours to settle out. The fuller's earth carries down with it all the impurities in suspension, and at the same time exerts a more or less bleaching action on the oil. The same quantity of fuller's earth may be used several times, especially if its function is simply that of a clarifying agent. Where however it is employed as a bleaching agent, it must be replaced with fresh material from time to time. The fuller's earth absorbs a large proportion of oil, which should be removed by treatment with benzoline or benzene before it is thrown away.

Where large quantities of oil are to be dealt with, it is a good plan to use fuller's earth combined with a filter press. The oil is heated with 2 to 3 per cent. of its weight of fuller's earth, as described above; then the oil is sent through a filter press, which removes the earth and the solid impurities in the oil. Special forms of filter press are made for dealing with oils.

Figure 43 represents a filter press made by Dehne of Halle, Germany, which is well adapted for pressing oils. These filter presses are easy to use, while they are efficient in operation.

It is quite possible when using a filter press to render oils, while not quite perfect, yet sufficiently so to be marketable, very quickly by sending the oil as it comes from the hydraulic press through the filter press. The oil comes out of the latter fairly bright and clear, and will find a ready sale. The solid matter which is filtered out is returned to the

kettle to be worked up with the next batch of seed. By this method of working there is produced nothing but oil and cake, no "foots" of any kind being formed.

Besides fuller's earth, there have been used in clarifying oils other solid matters, such as china clay, infusorial earth, etc., which act mechanically by carrying down the solid matter in suspension and absorb the moisture in the oil, and so leave the latter clear and bright.

Various processes have been devised for refining oils by

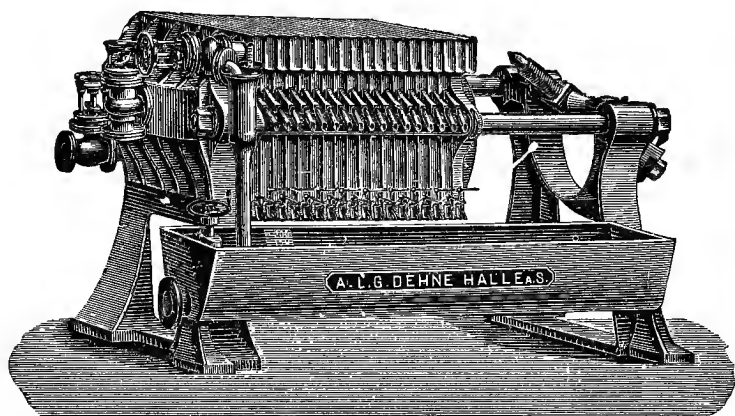


FIG. 43. Oil Filter Press.

chemical means; the two agents most commonly used being sulphuric acid and caustic soda. The general method of using these bodies will be detailed.

**REFINING OILS BY SULPHURIC ACID.** Sulphuric acid has a powerful action on all organic bodies, chiefly owing to its great affinity for water, which imparts to it dehydrating properties. As regards its employment in refining oils, its virtue depends on the fact that the extraneous vegetable matters found in crude oils are more easily acted upon by the acid than is the oil itself. Too much acid must not

however be used, or otherwise there is risk of it acting on the oil and thus leading to loss. The usual plan of treating oils is to place the oil in a suitable receptacle, a copper tank or iron pan, which should either contain a steam coil or, better still, be jacketed; means of agitating the oil and acid together should be provided, which may take the form of a mechanical agitator, or a current of air may be blown in during the operation. It is advisable for the bottom of the tank to be made conical for the purpose of a more effectual separation of the oil and acid, and a tap provided at the bottom of the cone for the purpose of running off the acid which collects. Many modifications of the process have been published from time to time. It will however be found that the details will have to be varied according to the oil which is being treated. Some oils come from the presses much purer than others. Such oils will require a less severe treatment than those which contain a large proportion of vegetable matter. Strong sulphuric acid should never be used; its action is too severe, and cannot well be controlled. It is always advisable to use a mixture of sulphuric acid and water—the proportion may vary according to the oil which is being treated—1 of acid to 1 of water is a very good ratio, while some oils may require an even weaker acid than this. Hartley recommends for linseed oil, 1 of acid to 2 of water; if the oil be very impure then the ratio 2 of acid to 1 of water may be used.

The oil to be treated is run into the tank and heated to about  $110^{\circ}$  to  $115^{\circ}$  F.; the latter temperature should never be exceeded, while it may happen that lower temperatures than  $110^{\circ}$  F. may be used. Then the mixture of oil, acid, and water is added with constant agitation and in a slow stream to the oil. The proportion used must vary according to circumstances, sometimes 1 per cent. of strong acid will be sufficient, in other cases 3 per cent. may be used. The



last-named quantity should however not be exceeded. It would be better to give the oil two treatments with a smaller proportion of acid. The oil and acid are thoroughly stirred together for about half an hour, then the mass is allowed to rest for twenty-four hours. At the end of this time 6 or 7 gallons of warm water at 150° F. for every 10 gallons of oil treated are mixed with the oil, and then the mass is allowed to stand for some days until a perfect separation of oil and acid liquor takes place. The oil is drawn off and washed again with water to free it from all traces of acid. The acid "foots," as they are called, are run away.

This method of refining is applicable to almost all seed oils, and is largely used in connection with linseed oil, rape oil, colza oil, nut oil, and also with fish oils. If it be used with any oils intended for lubricating machinery, it is necessary that the oil should be well washed with water to free it from all traces of the acid used in refining, which if left in might have a deleterious action on the metal of the bearings to which it is applied.

The acid treatment will not affect any free fatty acid which the oil may contain; any such will be left in the oil after the treatment is finished.

It has been recommended to use a strong solution (100° to 130° Twaddell) of zinc chloride, using from 1½ to 2 per cent. of the oil. This has no action on the oil itself, but it dehydrates it and coagulates all albuminous and vegetable matter the oil contains. It costs more to refine oils by zinc chloride than by sulphuric acid.

REFINING OILS BY CAUSTIC SODA. A good many oils are refined by using caustic soda, in fact some, cotton seed oil for example, cannot well be refined by other means, while to obtain certain qualities of oil an alkaline treatment to free the oil completely from acid constituents is necessary. Colza oil, for instance, is much used for illuminating

purposes ; if it contains any notable proportion of free acid it is rendered unsuitable for this purpose, hence an alkaline refining process is needful to be used with colza and other burning oils.

Alkaline processes not only free the oil from extraneous vegetable matter, but they remove any traces of resin, acid and colouring matters which the oil may contain, leaving a perfectly neutral and pure oil. Greater care is required in carrying out an alkaline process than is required for an acid process. This is due to the fact that, while the acid has no material action on the oil itself, and therefore little loss is likely to take place, the alkali has some action in the direction of saponifying the oil, and thereby a loss may occur. On the other hand, while the foots obtained with the acid treatment are useless, those obtained from the alkali process may be made use of.

The process is comparatively simple. The oil to be treated is run into a suitable vessel ; an iron tank serves very well. The requisite quantity of caustic soda lye is added, and the whole thoroughly agitated together for some time and then allowed to settle, an operation which may take some time, when two layers will form, one of a watery fluid containing much soapy foots, the other of clear oil. The watery fluid is drawn off into a tank, fresh weak alkali is run in, and the mass treated as before, after which the purified oil is well washed with water to free it from alkali.

The strength and proportion of alkaline solution used will depend upon the character of the oil to be treated. With all ordinary oils a lye of 8° to 12° Twaddell may be used. Crude cotton seed oil requires a stronger lye, one of from 15° to 20° Twaddell, while coconut oil can be refined with a lye of about 5° to 6° Twaddell. The quantity used will depend upon the amount of acidity of the oil which is being treated, the quantity of resinous matter it may contain, and the

amount of colouring matter. Hence few rules can be given as regards quantity of alkali to be used. Generally a half to one per cent. of caustic soda will suffice.

Occasionally trouble arises from the formation of emulsions which prevent the proper separation of the oil from the alkaline liquor. When this happens it is best to add a little solution of salt, sufficient to throw out the oil.

In some cases the oil has been treated with soda crystals melted by heat in their water of crystallisation. After being well mixed the mass is allowed to stand, when, as a rule, it easily separates into three layers, one of oil, the second of soapy matter, and the third of watery liquid.

When the oil is fairly free from mucilaginous matters, and is of poor quality in consequence of its containing much free fatty acid, this may be removed by agitating the oils with a weak solution of caustic soda or of carbonate of soda, but usually it will be found easier to treat them with milk of lime or with magnesia, followed by filtering from the lime or magnesia soaps which are formed.

In describing the various oils special note will be given to the methods of refining them.

In the section relating to olive oil will be found a description of an apparatus for separating oil from water.

*Oil Foots.* In the alkali method of refining oils a large quantity of "foots" is formed. These may be utilised in various ways according to their character. One very good method is to work them up for soap making in conjunction with other fats. Cases however occur where, owing to their being strongly coloured as in the case of cotton oil foots, this cannot be done. The best plan of dealing with such is to decompose the foots by weak sulphuric acid and distil the liberated fatty acids, etc. There is then obtained a distillate containing glycerine (from any undecomposed glyceride which may be present in the foots) and fatty acid. The

residue in the retort takes the form of "pitch," and will consist of the resinous matter present in the foots. The fatty acids which are obtained are not quite pure, but contain small quantities of hydrocarbon bodies produced by decomposition of the fatty matter by the heat employed in distilling.

Other processes for refining oils have been devised, but those just described are what are in common use in oil refineries.

#### BLEACHING OF FATS AND OILS.

As obtained by the processes described above, the oils and fats are often more or less coloured. In vegetable oils this colour is necessarily due to the presence of natural organic colouring matters, chlorophyll, erythrophyll, etc., present in solution in the oil. Linseed oil, brown rape oil, palm oil are examples of such oils which are strongly coloured. Very often in the processes of refining, such as have been described above, a large proportion of the colouring matters is removed, but traces of them will remain in the refined oils. Animal oils and fats are usually free from colour. Any such that may be present is generally due to exceptional circumstances.

There are a number of ways by which the colour can be removed from oils. A process which may give good results with one oil may not do so with another. In some cases it is sufficient to agitate the oil at a temperature of 120° F. with animal charcoal, followed by filtration. Blowing hot air through will in some cases (palm oil) destroy the colour. Air and light bleaching are sometimes resorted to, but in some cases it is necessary to make use of chemical reactions.

**BLEACHING BY HOT AIR.** By blowing a current of hot air at about 130° F. many fats and oils can be decolorised. Tallow, lard, and palm oil may be treated in this way. It

is not desirable that the action should be prolonged, or otherwise there is some risk of the oxidation of the oil. It is important that the air should be dry.

Figure 44 is a drawing of an apparatus made for bleaching palm oil by air devised by Messrs. Korting Bros. This consists of a cylindrical vessel of any convenient size to suit the quantity of fat that is being treated. K is a closed steam coil by means of which the fat can be heated up to

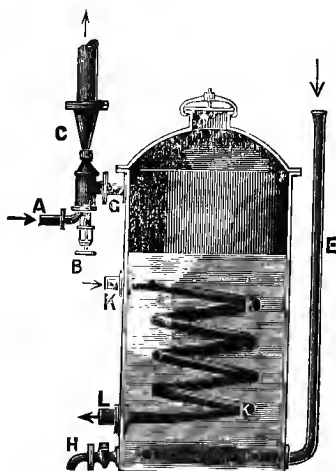


FIG. 44. Korting Brothers' Apparatus for Bleaching Palm Oil.

any required degree. E is a tube open at the top and terminating in a ring at the bottom inside the vessel, this ring having perforations. H is a draw-off valve. C is an injector worked by steam which enters at A. B is the regulating valve. By the action of the injector air is drawn from the upper part of the vessel and therein creates a vacuum which is filled by air being drawn in through E from the outer atmosphere and which rises in bubbles through the fat, bleaching

it. The operation is comparatively simple and is continued until a sample of the oil drawn from H shows that the bleaching has been carried far enough. It may be mentioned that the top of the vessel is fitted with a tight-fitting cover so that it may be hermetically sealed up.

**BICHROMATE OF POTASH PROCESS.** Watts has devised a process for the bleaching of oils by means of bichromate of potash, which is largely used, especially for bleaching palm oil. It is carried out in the following manner: The oil to be bleached is heated at about 120° to 130° F., then a quantity of bichromate of potash, about 22 to 28 lbs. per ton of fat, previously dissolved in a little water and thoroughly mixed with the fat, then hydrochloric acid to the extent of about 2 or 2½ per cent. of fat is added and also thoroughly mixed with the material. In the case of bleaching palm oil, the reddish orange colour changes first to a brownish green, and finally to a light green, the entire operation only taking a few minutes. Wet steam is now blown through for a few minutes, and then it is allowed to stand for some hours. The bleached oil is separated out and is skimmed off, and then washed with clean water to take out all traces of acid and chrome. Instead of hydrochloric acid, sulphuric acid may be used, but it does not give good results. This process can also be used for other fats and oils.

**CHLORINE PROCESS.** Chlorine is a powerful bleaching agent which may be employed for the purpose of bleaching fats and oils. It is necessary, however, that great care should be taken in using it on account of the fact that chlorine is a colouring agent in oils and fats, excess resulting in the formation of products which have deleterious effects upon them. The most convenient plan of working is to mix the fat with a solution of bleaching powder, using about 2 lbs. to 1 ton of fat or oil, then about three times the quantity of hydrochloric acid is added,

and the whole stirred together ; then the mixture is allowed to settle, the fat taken off, and the acid liquor run away. One advantage of the chlorine process is that it acts as a deodoriser to rancid fats. In the case with fats and oils which are strongly coloured, it is advisable to give them two treatments rather than to attempt to bleach them at one operation.

In place of using bleaching powder there may be employed potassium chlorate at the rate of 2 to 4 lbs. per ton of fat ; about twice the quantity of hydrochloric acid is added, and the operation done at a temperature of 150° F.

**SUN BLEACHING.** This is commonly carried out by exposing the oils and fats in colourless glass bottles to sunlight. From time to time the oil or fat is poured from one bottle to another with a view to exposing fresh portions to the action of the sunlight. This method is slow but is often employed, especially for castor and other oils used for medicinal or food purposes. The processes above described are those which are commonly employed in bleaching oils and fats. Different refiners, however, have various modifications of different processes, which they have been found to work well with the plant and appliances they have in use. Such modifications are often regarded as "trade secrets".

#### SOLVENT EXTRACTION PROCESSES.

All oils and fats are soluble in such bodies as ether, carbon bisulphide, benzoline, benzol, carbon tetrachloride, etc. This property is taken advantage of in both the laboratory and on the large scale for the purpose of extracting fats and oils from substances containing them. In the chapter on the analysis of fats and oils, descriptions of the methods used in the laboratory will be found.

Here will be described such methods as are used on a large scale. The principle on which all such apparatus works is that of treating the fat-containing substance with the solvent in a suitable vessel, then to run the solution into a still or retort and distil off the solvent by any means. The fat remains behind in the still, while the solvent is recovered and used over again. The best possible solvent is one that has great solvent properties for oils, can be distilled completely by means of steam, is free from odour and non-inflammable. The only substance among those named above which answers these conditions entirely is carbon tetrachloride, but unfortunately it is expensive.

Benzoline is commonly used for this purpose. It is light, has strong solvent action on oils, and is cheap. It has one disadvantage, it does not entirely volatilise by means of steam, consequently there is a tendency for a little of the benzoline to remain behind in the oil; again, it is very inflammable, and great care is needed in the working with this solvent. Carbon bisulphide is one of the best solvents to use. It is volatile at the temperature of boiling water. Being heavier than water it can be kept in tanks under water, thereby reducing the risk of danger from explosion or fire. It has unfortunately rather a noxious odour, which makes it unpleasant to work with. This odour is however largely due to impurities that are due to the materials from which the carbon bisulphide is made. By repeated use this odour becomes less offensive and unpleasant.

Benzol is an hydrocarbon obtained during the distillation of coal tar. It is freely volatile at the temperature of boiling water, and has strong solvent properties. It is highly inflammable, and therefore great care must be taken in working with it.



**DIETZ APPARATUS.** A very convenient form of apparatus for the extraction of oil by bisulphide is that of Dietz, as shown in Figure 45. This consists of an extraction tank B, in which is placed, between perforated plates at top and bottom, the material from which the oil is to be extracted; by means of a pump, carbon bisulphide, contained under water in the tank A, is passed through the extractor and so

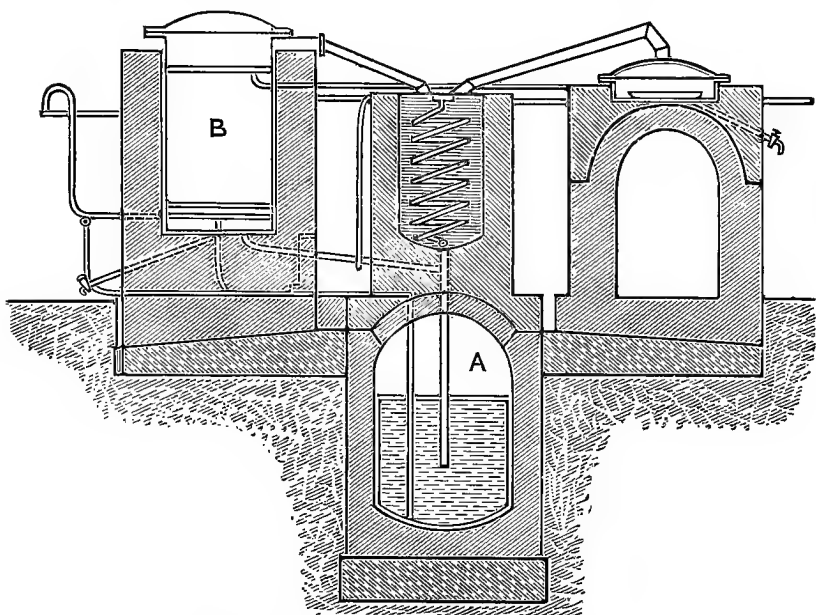


FIG. 45. Dietz Apparatus for Extracting Oils.

abstracts the oil from the material; from the extractor, the carbon bisulphide containing the oil flows into the still or retort D, where the carbon bisulphide is distilled off by steam, and is condensed in the coil condenser and flows back into tank A to be used over again. Residual oil left in the still D is run off from time to time by means of a discharge pipe. This plant is small and easy to work.

Figure 46 is a drawing of a small plant which can be made in any convenient size and used with any kind of solvent. It consists of three portions. First, the centre vessel which forms the extractor. The material is put in at the top, which is fitted with a lid which can be hermetically

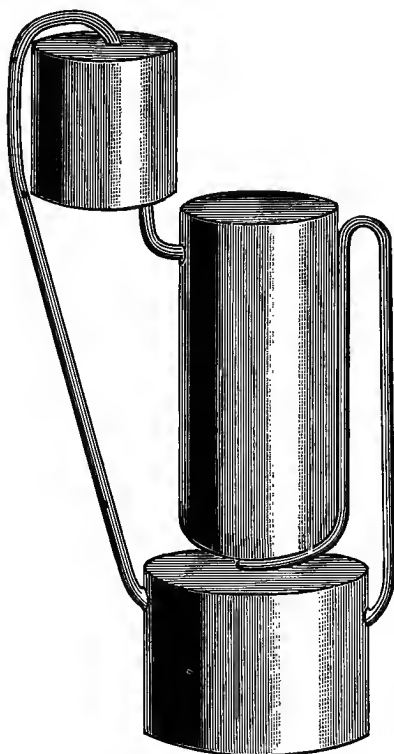


FIG. 46. Oil Extracting Plant.

closed. In this extractor there is also a perforated false bottom, on which the fatty materials are placed. Under this is a retort or still which can be heated by steam. This still communicates with the extractor by means of a tube, which proceeds from the bottom of the extractor and rises

up alongside it to nearly three-fourths of its height, and then turns down and enters into the still, ending near the bottom. From the top of the still a pipe passes to a condenser placed above the extractor, the end of the condenser worm passing into the top of the extractor. This plant is used in the following way: The fatty material is placed in the extractor and the required quantity of solvent in the still; by means of a steam coil the solvent is volatilised and passes into the condenser, where it is condensed, and flows as a liquid into the extractor; here it gradually accumulates, dissolving out the fat until its level rises above the level of the bend in the syphon tube, when it flows into the still; here it gets volatilised again and passes through the same cycle of changes. The fatty matter which it had in solution, however, is left behind in the still and can be run off from time to time, as required, through a discharge pipe placed on the bottom of the still.

Another method of extracting, using benzoline, is to have two large upright boilers side by side. Each is divided into three portions by partitions—the middle portion is the extractor and is provided with two manholes, one at the top for charging, the other at the bottom for discharging; the lower portion forms the still and is fitted with steam pipes; the top portion contains a condensing arrangement. This apparatus is used in the following manner. The extractors are filled with the oily material. Benzoline is placed in the still of one and is vaporised by means of a steam coil; the vapour, passing upwards into the condenser of the other boiler, is there condensed to a liquid and flows down through the oily material into the still, carrying with it the oil. When all the benzoline has been vaporised off from the original still, the action is reversed and the benzoline distilled back again; the oil it held in solution is, however, left behind in the still. The action is allowed to go on

until all the oil is removed from the raw material. This is then run off from the still to a storage tank.

The extraction of oils by means of volatile solvents is in itself very simple. The crushed seed or other oil-bearing material (dried fish, etc.) is placed in a closed vessel. Benzine, petroleum spirits, bisulphide of carbon, ether or any other suitable solvent is passed through it, and allowed to flow into a retort, carrying with it the oil in solution and leaving the exhausted substances in the extractor or macerator. The solvent in the retort is now distilled off and condensed for reuse, while the pure oil is left behind in the retort. So far the process is very simple, but there are many difficulties: one consists in recovering the solvent from the waste product in the extractor. This part of the apparatus consists essentially of a large tank, with inlet and outlet pipes for the circulation of the solvent, and steam connections to drive off the residual solvent after the material in it has been exhausted.

These extractors must necessarily be of a considerable size, and when extraction has been completed, there is left behind a large bulk of residual material, starch and husk (in the case of seeds), saturated with solvent which ought to be distilled off before the extractor is emptied. This is necessary for two reasons. First, because the manufacturer could not afford to lose all this solvent; secondly, because its vapours are inflammable, and might if allowed to escape freely become a source of danger. And herein lies the chief difficulty: the material left in the extractor is a bad conductor of heat, and therefore it is very difficult to heat the mass, when in large bulk, sufficiently throughout to ensure complete volatilisation. To this a second difficulty is added, by the fact that the volatilisation of the solvent has the tendency of greatly depressing the temperature in those parts which are not easily reached by the heat of the steam.

This difficulty is only partially overcome by admitting steam into the extractor, as what volatilises in the lower part condenses again in the upper layers, until the whole mass is sufficiently heated throughout, which, on account of the non-conducting properties of the material, is achieved after many hours only. But when the solvent has to be thus driven off by live steam, the material is being cooked at the same time and when taken out is in a moist state, which in many cases is detrimental to what otherwise would be a valuable product.

The residual meal cannot be stored in this condition, as it soon spoils, and is only fit for manurial purposes.

The apparatus shown in Figure 47 was designed to overcome all these defects. The difficulty arising out of the unmanageableness of the material while in bulk has successfully been conquered by treating small and successive quantities at one time. This is being done by practically reversing the older process. That is, instead of passing the solvent through a large mass of material, here the material to be extracted is passed through the solvent, the latter flowing at the same time in the opposite direction.

In what manner this is effected will be best understood by a reference to the accompanying drawing. C, C2, C4, C5, and C6 are cylinders alternately communicating with each other at top and bottom, in which are working screw carriers. The material is fed in at A (which, when not at work, is hermetically closed by a water seal) and carried forward by a screw working in B, and falls down the vertical shoot into C. Here it is carried upwards by a screw to C1, where the first cylinder communicates with the second and falls on to the screw working in C2, is carried downwards and falls through communicating channel C3 on to the next screw. This again carries the material in an upward direction and to C4, whence it is carried downward in C5,

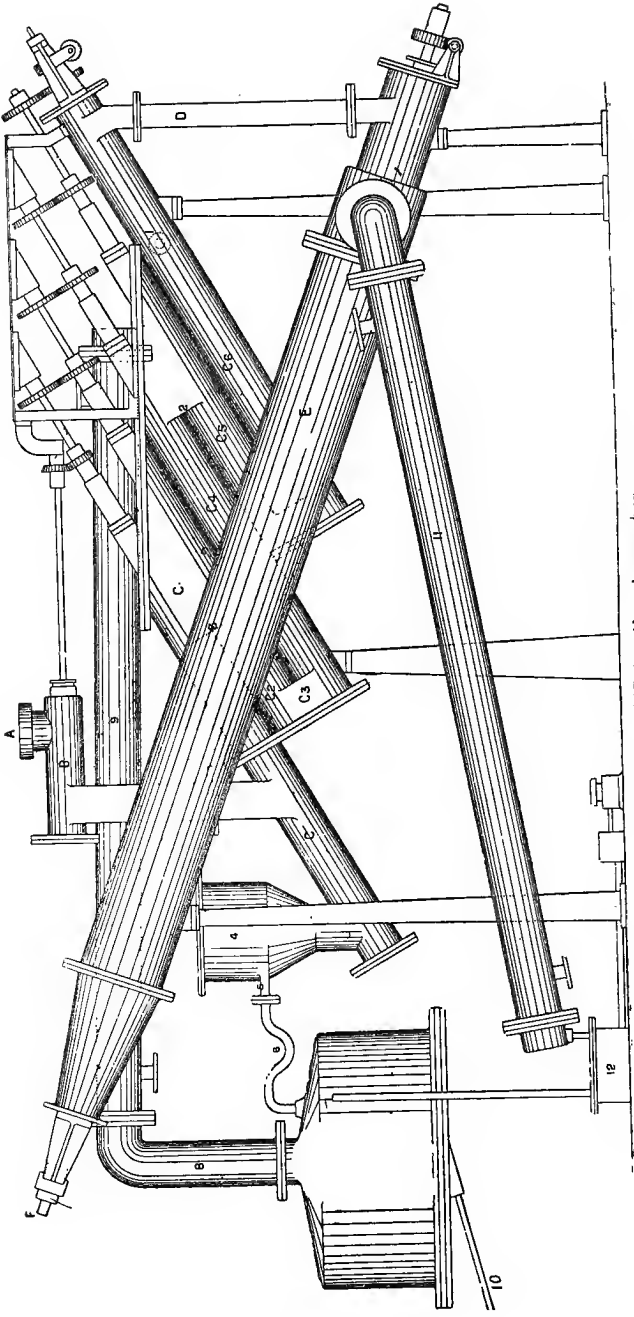


Fig. 47. Oil Extracting Apparatus.

up again in C6, in the upper part of which there is a squeezing arrangement to express the excess of solvent, and falls then through D into kiln E, which is provided with a steam jacket and forward screw, where the exhausted material is deprived of the residual solvent. This is easily effected, as the material is spread out in a thin layer over a large heated area. The vapours, which are considerably heavier than the air, and hence have a downward tendency, are allowed to escape at the lower end of the kiln, are condensed in their passage through the condenser 11, collected in receiver 12, and pumped back into the machine at 1. While the material thus travels from left to right, the solvent flows in an opposite direction, whereby the partially extracted meal, etc., meets in each successive cell with a purer stream of solvent, thus ensuring nearly complete exhaustion. On the other hand the solvent comes successively in contact with material richer in oil, finally passing into the first cell (the last as regards the direction of the solvent), where it meets the largest quantity of oil, passes through a filter H, and thence into a retort. This latter part of the apparatus is provided with several trays, each steam jacketed, over which the mixture of oil and solvent flows in a thin stream; the solvent being volatilised and condensed in 9, while the oil flows out in a continuous stream at 10. The apparatus is sealed by water throughout.

### SECTION E.

#### ANIMAL AND VEGETABLE LUBRICATING OILS.

##### TALLOW.

Tallow is or should be derived from the fat of cows, oxen, sheep, goats, and similar animals, the best fat being taken from the thicker deposits which surround the abdomen, known generally among butchers as the skin, although it is known by other names in some places. Anatomists call it the *omentum*. Tallow is also obtained from the fat sur-

rounding the large muscles, the kidneys, and the other organs of the body. Fatty matter can be and is obtained from other parts of the animal, such as the intestines, bones, etc., and such fat is often sold as tallow; sometimes with a qualifying distinction as bone tallow; at other times, and this wherever possible, without such a qualifying description. Whenever tallow is dealt in as tallow, it is always understood to be the fat obtained from the parts above-named, and the sale of any other kind of fat should be regarded as a fraud upon the buyer.

The deposit of tallow as it exists in the animal body is found to be contained in small cells or bladders of animal tissue. The reason for its being in this condition is that while being in fluid so as to move with every motion of the parts, yet it is not able to flow from the spot in which it is found. As it is taken from the body, the crude fat is known as butcher's fat, and is usually purchased from the butchers by the tallow refiner, who, if he be located in a large town, often distinguishes between "town fat" and "country fat," the former being usually fresher, and therefore yielding a better quality of tallow than the latter. The idea or principle which underlies all processes for the extraction of the tallow from this rough fat is to separate out by some means the animal tissue from the actual fatty matter, a process which is known as rendering. The various means which have been adopted to do this have been described, p. 128 *et seq.*

Of fatty matters obtained from other parts of the animal body, the intestines give what is called "tripe tallow," the feet yielding "neatsfoot oil". The processes for extracting these greases or fats do not differ essentially from those described above, but, as a rule, they are obtained by simply boiling the various parts of the animal in water, and skimming the fat which is obtained from the top of the water where it collects, whilst the tripe and feet are used as food.



A great deal of fat largely used in making soap is now extracted from bones, and sold as "bone grease," "bone fat," "bone tallow," and not seldom, wherever possible, as tallow. All bones do not contain tallow or fat; the best are cows' and bullocks' shank bones, which are hollow, and contain a fairly large proportion of good fat, often separately extracted and sold as "marrow tallow". The more solid bones found in the animals do not contain much fat, and scarcely pay for its extraction. Horses' bones contain so little fat they are not worth the process of treatment. In dealing with bones it is therefore worth while to sort them into those which are worth treating for the fat and those which are not worth so treating. The method of extraction is given on p. 137.

Tallow comes into this country from all parts of the world. Now it is a well-known fact with regard to natural products obtained from various localities that they vary somewhat in appearance, colour, odour, and consistence, etc., to say nothing of minor differences in chemical composition. Tallow is no exception to this rule, hence in the tallows which are sent into England from North America, Australia, Russia, India, etc., there are certain minor differences by which experts can tell the locality from which the tallow came. There can scarcely be any doubt but what these differences are caused by the character of the food on which the cattle feed in the various localities, and which must vary to a great extent. Commercially, tallows are distinguished according to their country of origin, and of the cattle—oxen or sheep, or into beef or mutton—from which they are obtained. Russian tallow comes chiefly from Cronstadt, Odessa, Taganrog, and St. Petersburg. It is derived chiefly from oxen, and is a hard, yellowish tallow, better suited for candlemakers than for soapmakers. A large proportion of the Russian tallow finds its way from Siberia, but no distinction is made

between this tallow and that from other parts of Russia. South America sends both "beef" and "mutton" tallow. It is chiefly shipped from the River Plate ports. It is of a strong yellow colour, and usually of fair quality, and serviceable for all uses. North American tallow is of very good quality, rather paler in colour than South American, and is the favourite tallow of soapmakers. It is mostly "beef" tallow that comes from North America, but "mutton" tallow is also sent over. Australia also sends large quantities of both "beef" and "mutton" tallow to England of fairly good colour and quality. The value of the tallow depends on its consistence—the harder the tallow and higher its melting point, the more valuable it is. These points vary very much within certain limits, which will be presently pointed out.

The chemical composition of tallow varies somewhat according to the method of feeding and the locality as well as the kind of tallow. Tallow consists essentially of the two glycerides, olein and stearin, the latter predominating, forming from 60 in soft to 80 per cent. in hard tallows. Margarine is also probably present in some tallows, while there are also nearly always small quantities of animal tissue, colouring matter, water; but these should not altogether amount to more than from  $\frac{1}{2}$  to 1 per cent. of the total. Beef tallow contains more olein than mutton tallow, so that it is rather softer in consistence, and therefore better adapted for soapmaking and lubricating and for making tallow oil, whereas mutton tallow is more suitable for the candlemaker. The specific gravity of beef tallow ranges from 0.935 to 0.939, while that of mutton tallow ranges from 0.937 to 0.940 at 60° F. (15° C.); while at 212° F. (100° C.) the specific gravity is from 0.860 to 0.862. The melting point of tallow varies very considerably, usually ranging from 36° to 49° C. (97° to 120° F.)—the lower limit

is that of a soft tallow, while the higher limit is a hard tallow.  $39^{\circ}$  C. ( $102^{\circ}$  F.) is the average melting point of tallow. After being melted it begins to solidify at rather lower temperatures, from  $33^{\circ}$  to  $46^{\circ}$  C. ( $115^{\circ}$  F.), but at the moment of solidifying the temperature rises a few degrees. When pure, tallow should be white, fairly firm, and without much odour and taste. It is soluble in from 40 to 44 times its volume of alcohol. Generally, it contains a small quantity of free acid, ranging from 0.75 to 7 per cent., although occasionally samples with larger quantities are met with. For soapmaking, the presence of free, fatty acid is not detrimental, but rather otherwise; but for lubricating machinery it is decidedly disadvantageous.

When melted tallow is allowed to cool very slowly at a temperature of not less than  $27^{\circ}$  to  $30^{\circ}$  C. ( $80^{\circ}$  to  $86^{\circ}$  F.), it forms a granular mass, the stearin crystallising out in the form of small nodules which can be separated out from the more fluid mass by pressure. The process is known as "seeding," and is largely applied to the separation of the stearin of the tallow for use in making candles, while the liquid which passes through the press is known as "tallow oil," and is used for lubricating machinery and soapmaking.

When boiled with caustic alkalies, tallow is converted into soap. Of caustic soda, tallow usually takes about 13.79 to 13.85 per cent. to completely saponify it, while of caustic potash it requires 19.32 to 19.38 per cent. The alkalies being in both cases taken as being chemically pure, of the ordinary commercial products more will be required, according to the strength of the article, which varies very much. When the soap formed by boiling tallow and alkali together is treated with acid, the fatty acids of the tallow are separated and usually are found to amount to 95 per cent. of the tallow used. The melting point and specific gravity of these fatty acids vary with the quality of the tallow. (See page 179.)

Tallow is frequently adulterated. Among other bodies have been used soft fats from other parts of the animal, such as bone or tripe tallows, cottonseed oil, seal oil, stearin from wool grease, among fatty matters, to say nothing of china clay, starch and similar products. It is by no means an easy matter to detect some of these adulterants: cottonseed oil stearin is very difficult to detect. The specific gravity of the fat at 100° C., the melting point, is also some guide, while the large proportion of solid fatty acid would be a clue to its addition. Cottonseed oil can be detected by its reducing the specific gravity, melting point, and increasing the proportion of liquid fatty acids, as well as by the silver nitrate and iodine test. Stearin from wool grease can be detected by the tallow containing a large proportion of fatty acid, as well as by the silver nitrate test. Bone grease can be detected by the tallow containing phosphate of lime, which is a characteristic ingredient of bone grease. The addition of such matters as china clay and starch can be detected by melting the tallow and allowing these insoluble matters to settle out. Such forms of adulteration are now rare, and show unskilful work on the part of the adulterator. Paraffin wax and scale and mineral oil are sometimes added; these may be detected by their reducing the percentage of potash required to saponify the tallow, by the sample having a low flash point (under 400° F.), while the amount can be ascertained by the test described in the chapter on Oil Analysis.

Tallow is now rarely used for lubricating machinery. At one time it was largely used for lubricating steam engine cylinders, but it has been superseded by the petroleum cylinder oils (page 106). It is used now in small quantity for lubricating heavy engine bearings—bearings of rolling mills—and for making lubricating greases.

## CONSTANTS OF TALLOW.

Specific Gravity at 15° C. (60° F.)	. . .	0.943 to 0.942.
„ „ „ 50° C. (122° F.)	. . .	0.895.
„ „ „ 100° C. (212° F.)	. . .	0.862.
Melting Point, 42° to 46° C. (107° to 115° F.).		
Solidifying Point, 36° C. (98°).		
Insoluble Fatty Acids (Hehner Value),		95 to 96 per cent.
Saponification Value (Koettstorfer Test),		19.3 to 20 per cent.
Iodine (Hubl Test),		39 to 44 per cent. (absorbed).
Reichert Value, 0.25 cc. $\frac{N}{10}$ KOH.		
Viscosity at 120° F.	. . . . .	53.
„ „ 150° F.	. . . . .	35.
„ „ 212° F.	. . . . .	25.

## CONSTANTS OF FATTY ACIDS FROM TALLOW.

Specific Gravity at 100° C. (212° F.),	0.8698.
Melting Point, 43° to 44° C. (108° to 110° F.).	
Solidifying Point, 42° to 43° C. (107° to 108° F.).	
Molecular Weight (Combining Weight),	284.
Iodine Value,	40 per cent.

## TALLOW OIL.

Tallow oil is obtained from tallow by melting and keeping the tallow in a warm room at about 80° to 90° F. for some hours. The stearin it contains crystallises out in the form of small granular particles; hence this process is known as "seeding". The seeded tallow is then placed in canvas cloths and put under hydraulic pressure, when the olein it contains comes out together with a little of the stearin and forms the tallow oil of commerce. It is also sold under the names of ox oil, animal oil, etc. Its chief use is for lubricating machinery, but it is used for making soap—when a white soap, rather soft and more soluble than a tallow soap, is required. Tallow oil varies much in consistence. Some samples are more fluid than others. This depends upon the proportion of stearin which the oil contains. If there is much, then the oil is solid; if little, then the oil is liquid. The specific gravity varies for the same reason from 0.911 to

0.915. Tallow oil contains a varied proportion of free acid from none in well-prepared samples to 15 to 16 per cent. in others. Tallow oil should be quite white when cold, or have at the most a faint yellow tint. When melted, it ought to be quite clear and bright, free from any cloudiness or broken particles of any kind. It has only a very slight odour of an animal fat.

It is employed as a lubricating oil for shafting and heavy bearings, and is often mixed to the extent of 10 per cent. with mineral oils to form spindle oils and loom oils, for which it answers very well.

Tallow oil has a viscosity at 100° F. of 65, at 150° F. of 32, and at 212° F. of 23. Flash point, 475° F. to 500° F. varying with the quality.

#### LARD OIL.

Lard oil, like tallow oil, is not much used in soapmaking, finding its principal use in lubricating machinery. It is prepared from lard in the same way as tallow oil is made from tallow. It resembles the last oil in its properties and uses. It is if anything rather lighter in colour and has less odour, which resembles that of lard. It is used in lubricating machinery, chiefly for mixing with mineral oils in the preparation of spindle and loom oils, usually  $7\frac{1}{2}$  to 10 per cent. being added. It works well.

#### NEATSFOOT OIL.

This oil is obtained from the feet of cows and sheep. These are skinned, then boiled in water, when the fatty matter they contain collects on the surface of the water and is skimmed off from time to time and sold as neatsfoot oil.

Occasionally it is subjected to a purifying process which consists in boiling it up with water.

Neatsfoot oil makes its appearance either as a turbid

or a limpid oil. In winter time it will become solid from deposition of stearin. Its odour is pleasant, its taste sweet. Its properties are practically identical with those of other animal oils obtained from the cow or sheep, and it is often difficult to distinguish between such oils.

The specific gravity is usually about 0.912. It takes 19 per cent. of caustic potash to saponify it.

Neatsfoot oil was at one time largely used for lubricating steam engine cylinders, but its use for this purpose is now limited. It is not used by itself as a lubricating oil, but is often mixed with hydrocarbon oils to form loom, spindle, or shafting oils. Its lubricating powers are good, and as it is fairly free from free acid it does not corrode the bearings.

#### VEGETABLE OILS.

##### PALM OIL.

Palm oil is obtained from the fruit of various palm trees, natives of the oil regions of the West Coast of Africa. The tree which yields the largest proportion of the palm oil of commerce is the *Elais Guinensis*. The fruit of the palm is about the size of a small plum and hangs in bunches from the trees. Like the plum it contains an outer pulpy mass or pericarp and an inner kernel. From the outer pulp is obtained palm oil, while the kernel yields palm nut or palm kernel oil, also used in soapmaking, but which has different properties to palm oil.

The natives of the oil regions employ a variety of methods for the purpose of extracting the oil from the fruit. The commonest plan consists in stacking the nuts as they are taken from the trees in heaps for from seven to ten days, when, by the composition of some of the vegetable tissue surrounding the husks of the nuts, the husk can be readily removed, leaving the internal pulp and kernel. The pulp is of rather a hard nature, and to soften it the

nuts after being husked are thrown into pits and plantation leaves covered over them then with earth and palm leaves. In this condition they remain for a period varying from three weeks to three months, according to the fancy or practice of the particular tribe of Africans who are making the oil. At the end of the period named the pulp will have been converted into a soft mass. It is now thrown into pits lined with stones, where it is subjected to a pounding process whereby the pulp is separated from the kernels. The former is now thrown into boiling pots and boiled with water, when the oil rises to the top and is skimmed off, any vegetable tissue which may accompany the pulp passing into the water; or the oil may be separated from the vegetable by heating it with the water so as to melt the oil, and then squeeze the mass into bags when the oil flows out. The quality and consistence of the product depend partly on the particular species of palm from which it is made, but as to this point definite information is lacking; but more particularly upon the care with which the process of extraction has been carried out, and the length of time the fruit is allowed to remain in the pits. A long steeping results in the oil being harder, but at the same time its quality is materially decreased, it acquires a rancid odour, its colour is not so bright, and it contains much free acid indicating that a decomposition into acid and glycerine has taken place. A short steep gives an oil of a sweet odour and a bright colour. The process of extracting palm oil being as crude as it can well be, it is evident that the commercial article is far from being all pure fat. It must contain some trace of vegetable tissue, etc., which being very liable to ferment will in course of time gradually bring about the decomposition of the oil, resulting in its becoming more or less rancid, and losing its odour and colour.



Palm oil is a solid oil about the consistence of butter. It has an orange to golden yellow colour, which is highly characteristic but is liable to vary very much. Salt Pond and Brass oils have usually a brownish colour, Lagos oil is a bright orange, Sierra Leone is rather redder, New Calabar oil is a golden yellow; the colour is probably dependent upon the species of palm from which the oil is obtained in the first instance, and partly on the process of extraction. Lagos oil is the best and most neutral quality, the proportion of free acid it contains rarely exceeding 10 per cent., and the amount of water and other impurities not more than 2 per cent., and in consistency it is the softest of palm oils. Brass, so far as impurities are concerned, comes next to Lagos oil. It is the hardest of the palm oils, and on that account is the quality most in favour with candlemakers. It usually contains a large percentage, 53 to 65, of free fatty acid, and by far the largest proportion of palmitic acid of any variety; hence its hardness. Salt Pond palm oil is one of the worst varieties of palm oil to be found in the English market, the amount of impurities often being found to amount to 20 per cent.; while the free acid has been found by Norman Tate to be as high as 80 per cent., indicating that little actual oil is present. The colour and odour are usually poor. Half Jack, Bonny, New Calabar oils occupy intermediate positions between these oils in hardness and quality generally. Palm oil has a peculiar and violet-like odour, which is communicated to the soap which is made from it.

Chemically, palm oil consists of a mixture of palmitin and olein in various proportions, with varying quantities of free palmitic and oleic acids. The specific gravity of palm oil at 15° C. varies from 0.920 to 0.926. The specific gravity at 100° C. ranges from 0.857 to 0.859. The melting point is

exceedingly variable, ranging from 25° to 36° C. (77° to 97° F.), the solidifying point being a few degrees less. When saponified palm oil yields from 94 to 97 per cent. of fatty acids, the setting points of which range from 41° to 46° C. (106° to 113° F.), the combining equivalent from 273 to 274. Palm oil takes from 19.6 to 20.2 per cent. of caustic potash, KOH, or from 14 to 14.4 per cent. of caustic soda, NaOH, to saponify it.

Palm oil is mostly used in the form of greases in the lubrication of machinery. (See chapter vii.) It is rarely used by itself or in admixture with mineral oils as is tallow. The great variation in its quality and the corrosive action of any free acid it may contain on the bearings of machinery are obstacles to its use.

#### CONSTANTS OF PALM OIL.

Specific Gravity at 15° C. (60° F.)	. . .	0.920 to 0.924.
"    "    "    50° C. (120° F.)	. . .	0.893.
"    "    "    100° C. (212° F.)	. . .	0.8586.
Melting Point from 27° to 42° C. (80° to 107° F.).		
Hehner Value (Insoluble Fatty Acids), 96.5 per cent.		
Saponification Value (Koettstorfer Test), 20.2 per cent. KOH.		
Reichert Value, 0.5 cc. $\frac{N}{10}$ , KOH.		
Iodine Value (Hubl Test), 51 to 52 per cent.		
Viscosity at 100° F.	. . . . .	55.
"    "    120° F.	. . . . .	34.
"    "    150° F.	. . . . .	28.

#### CONSTANTS OF FATTY ACIDS FROM PALM OIL.

Specific Gravity at 100° C. (212° F.), 0.8369.
Solidifying Point, 45.5° C. (113.5° F.).
Melting Point, 50° C. (122° F.).
Saponification Value, 20.6 per cent. KOH.
Combining Equivalent (Molecular Weight), 270.

#### PALM NUT OIL.

The nuts or kernels of the fruit are collected and imported in large quantities into this country for the purpose of press-

ing the oil from them. In some places a very crude method is in use for the extraction of the oil. The nuts are put into a pan over a fire and charred slightly, some of the oil exudes and is poured off; the roasted nuts are now ground and boiled with water; the oil they contain rises to the top and is skimmed off; after a short boil the mass of kernel meal is again ground up, mixed with a little water, and mixed up again and boiled up, when more oil is obtained. This is skimmed off as before. The oil obtained by this process is of a dark colour.

Palm nut oil is of a white or faintly yellowish colour, of consistency similar to butter. In odour it resembles coconut oil, from which it can hardly be distinguished. The melting point ranges from 26° to 30° C. (78° to 86° F.). Much depends upon the proportion of oil obtained from the kernel. The first portions which are pressed out are the softest, and have the lowest melting point; the last portions are hardest, and have the highest melting point. It takes from 22 to 24 per cent. of caustic potash, or from 15½ to 17 per cent. of caustic soda to saponify it. In this respect it resembles coconut oil, to which it approximates in composition by containing a large proportion of the lower fatty acids, lauric, capric, capryllic, and caproic acids, but not to so great an extent as does coconut oil. Oudemans gives the following analysis of palm nut oil:—

Glyceride of Oleic Acid . . . . .	26·6
Glycerides of Stearic, Palmitic, and Myristic Acids . . . . .	33·0
Glycerides of Lauric, Capric, Capryllic, and Caproic Acids . . . . .	40·4

Palm nut oil is fairly free from free acid, and is not liable to become rancid readily. In its specific gravity at both 60° and 212° F. it closely resembles coconut oil.

Palm nut oil is largely used in soapmaking in the place of coconut oil, but rarely in lubricating machinery.

Palm nut oil is adulterated with lard, tallow, and other

cheap fats and oils. Such adulteration can be detected by means of the saponification value and the distillation test, which will suffice to detect any adulteration.

#### CONSTANTS OF PALM NUT OIL.

Specific Gravity at 15° C. (60° F.) . . . .	0.952.
"    "    "    40° C. (105° F.) . . . .	0.9119.
"    "    "    100° C. (212° F.) . . . .	0.8731.
Solidifying Point, 20.5° C. (71° F.).	
Melting Point, 27° to 28° C. (79° to 80° F.).	
Hehner Value, 91.1 per cent.	
Saponification Value (Koettstorfer Test), 24 per cent. KOH.	
Reichert Value, 2.4 cc. $\frac{N}{10}$ KOH.	
Iodine Value, 10 to 13 per cent.	

#### CONSTANTS OF FATTY ACIDS OF PALM NUT OIL.

Solidifying Point, 20° to 23° C. (71° to 76° F.).
Melting Point, 25° to 28° C. (77° to 80° F.).
Saponification Value, 25 to 26 per cent. KOH.
Combining Equivalent (Molecular Weight), 211.
Iodine Value, 12.07 per cent.

#### COCONUT OIL.

Coconut oil is the product of the familiar coconut, or cocoa nut, as it is sometimes called, the fruit of the coco palm—*cocos nucifera*.

The fruit of this palm is a very useful product. It is of large size; the outer portion consists of a fibrous mass which is made into ropes, mats and carpets. Inside this is the nut proper, consisting of a hard outer portion only useful as fuel. Inside this is a layer of pulpy matter of a white colour, which is that portion of the fruit patronised by the young idea at fair times. The central portion of the fruit is occupied by a milky fluid. The pulp is of a very oily nature, the proportion of oil usually averaging over 50 per cent. This pulp is dried by exposure to the air, and in that condition is known as "coprah," and is imported into this country in large quantities for the purpose of extracting the oil out of

it. The native method of extracting the oil consists of heating the pulp with water, when the oil rises to the top and is collected. Another method commonly followed when inferior qualities of oil are wanted is to heat the pulp with a little more water so as to render the oil more fluid, and then to subject the mass to pressure in a rude kind of oil-press worked by oxen. In some places the pulp is dried and then grated by means of cutting machines, and this, after being heated with water, is pressed, yielding a large quantity of a good quality of oil.

Coconut oil comes into this country from many places in South Eastern Asia. The best is that from Ceylon; Cochin China oil ranks very close behind; Malabar oil is of a very good quality; Manilla oil is good; some comes from Mauritius and the Fiji Islands.

In this country coconut oil is pressed from coprah.

Coconut oil makes its appearance in this country in the form of a white but soft fat. In the Asiatic countries it is a water-white, rather limpid oil. It melts at from  $20^{\circ}$  to  $25^{\circ}$  C. ( $68^{\circ}$  to  $77^{\circ}$  F.); its specific gravity at  $60^{\circ}$  F. is 0.931; at  $212^{\circ}$  F. it is 0.870. Its odour is pleasant and characteristic, resembling that of the coconut. It is liable to become rancid, when its odour becomes more pronounced.

It is fairly easily saponified, especially by strong alkaline lyes, and on that account it is much employed in the manufacture of soaps by the cold process. It takes from 24.5 to 26 per cent. of caustic potash, or from 17.4 to 18.7 per cent. of caustic soda, to completely saponify coconut oil, a larger amount than is required for any other known fat.

Coconut oil is one of the most complex oils known as far as regards its chemical composition. The principal fatty acid present is lauric acid,  $\text{HC}_{12}\text{H}_{23}\text{O}_2$ ; there is also present caproic acid,  $\text{HC}_6\text{H}_{11}\text{O}_2$ . These acids are all soluble in water, and are volatile when distilled with steam or water.

It is the presence of these acids which causes coconut oil to require so much alkali to saponify it and to form a soap which works with hard water, for the lime salts of these fatty acids are, comparatively speaking, soluble in water, and the alkali soaps are much more freely soluble in saline solutions than is the case with alkali salts of oleic or stearic acids for example. There are also present in coconut oil stearic acid,  $\text{HC}_{18}\text{H}_{35}\text{O}_2$ ; palmitic acid,  $\text{HC}_{16}\text{H}_{31}\text{O}_2$ ; myristic acid,  $\text{HC}_{14}\text{H}_{27}\text{O}_2$ , with smaller quantities of other acids of the same series. There are but comparatively small quantities of the acids of the oleic series. Of course it should be understood, in speaking of fatty acids in coconut oil, that these do not exist as free acids, but in the form of glycerides. Lauric acid has a combining weight of 200. The combining weight of the fatty acids which can be extracted from coconut oil by saponifying and liberating the acids with sulphuric acid ranges from 196 to 204. When these fatty acids are distilled with water, the distillate possesses an acid reaction due to the volatile or soluble acids coming over: the acidity from 100 parts of oil is equal to 0.78 to 0.83 of caustic potash. Butter and palm nut oil have a similar composition. Coconut oil has very little power of absorbing iodine or bromine; of the former it takes up 8.9 to 9 per cent., of the latter 5.7 per cent. It yields from 12 to 13 per cent. of glycerine, and from 94 to 96 per cent. of fatty acids.

Coconut oil is rarely adulterated, and then chiefly with animal fats and greases, with solid vegetable fats and stearins. Any such adulteration would not be very difficult of detection. The odour, alteration of the saponification value, reduction of the specific gravity at  $212^\circ \text{F}$ ., and reduction of the amount of volatile acids will suffice to detect such adulteration.

Coconut oil is used in India and other eastern countries for lubricating machinery and gives very good results,

especially on light-running machinery. In this country it is often used, mixed with hydrocarbon oil, as a spindle or loom oil, for which purpose it works well.

## CONSTANTS OF COCONUT OIL.

Specific Gravity at 15° C. (60° F.) . . . . .	0.930.
"    "    "    40° C. (105° F.). . . . .	0.9115.
"    "    "    100° C. (212° F.) . . . . .	0.8736.
Solidifying Point, 16° to 20° C. (60° to 70° F.).	
Melting Point, 20° to 28° C. (70° to 80° F.).	
Saponification Value (Koettstorfer Test), 25 to 26 per cent. KOH.	
Hehner Value (Insoluble Fatty Acids), 83 to 88 per cent.	
Reichert Value, 8.5 cc. $\frac{N}{10}$ KOH.	
Iodine Value (Hubl Test), 8.9 to 9.3 per cent.	
Viscosity at 100° F. . . . .	40.
"    "    120° F. . . . .	27.
"    "    150° F. . . . .	22.

## CONSTANTS OF FATTY ACIDS FROM COCONUT OIL.

Specific Gravity at 100° C. (212° F.), 0.8354.
Solidifying Point, 20° C. (70° F.).
Melting Point, 24° to 25° C. (75° to 77° F.).
Combining Equivalent (Molecular Weight), 196 to 206.
Iodine Value (Hubl Test), 9.3 per cent.
Iodine Value of Liquid Fatty Acids, 54 per cent.

## CASTOR OIL.

Castor oil is obtained from the seeds of the castor oil plant, *Ricinus communis*, a native of India, where it grows luxuriantly. The plant is a pretty common one in English conservatories, and so is familiar to most persons. The seeds are of a comparatively large size, and of a greyish colour and very lustrous. They contain a large proportion of oil, nearly 50 per cent., which is extracted by pressure in the usual way, or by boiling the seeds in water. Several qualities are recognised. That first extracted by pressure is the best, and sold chiefly for pharmaceutical purposes; the lower grades for lubricating oil and soap, and the average

commercial qualities are imported from Calcutta, Madras, Bombay and France. What is known as first pressure, French, is about equal in quality to what is known as second quality, Calcutta.

Castor oil is a thick, viscid oil. In colour, it varies from colourless in the pharmaceutical product, to a greenish yellow in the poorer sorts. Its specific gravity ranges from 0.960 to 0.970; the average being 0.964. Occasionally samples are met with having a specific gravity below 0.960, but such are rare. The odour varies considerably; the best qualities are fairly free, but the poorer sorts have a nauseous odour. The taste also varies in the same way; the common qualities have a peculiar nauseous taste, from which the best are free.

It does not begin to become solid until a temperature of 18° C. (0° F.) is reached, and even then only a few flakes are deposited. This oil is distinguished from other fatty oils by its peculiar physical and chemical properties; it has a very high specific gravity, a high viscosity. The relative viscosities of castor and sperm oil are as 1248 and 58.5 respectively, at 70° F., which figures will convey some idea of the viscid character of the oil. It is readily soluble in alcohol, 1 part in 4 of rectified spirits at 15° C. (60° F.). This enables an addition of other oils to be detected. It is insoluble in petroleum spirits or mineral oil at ordinary temperatures. On being heated, castor oil will mix with, or become soluble in, the petroleum spirit or mineral oil, but as the temperature cools down again the two liquids separate out.

Castor oil consists of a little palmitin which separates out when the oil is cooled down, and the glyceride of a peculiar (ricinoleic) acid, which has hitherto only been found in castor oil. This acid has the composition shown in the formula  $C_{17}H_{32}OHCOOH$ . It differs from other fatty acids in containing three atoms of oxygen, and there is reason for thinking that this extra atom of oxygen is combined with an



atom of hydrogen in the form of hydroxyl, as shown in the formula given above. Ricinoleic acid is therefore an hydroxyl fatty acid. The presence of this hydroxyl group gives to ricinoleic acid the property of forming, with sulphuric acid, ethers. On this property is based the use of castor oil in the preparation of olein oil for calico printers' use. Castor oil yields about 9.1 per cent. of glycerine and 96.1 per cent. of fatty acids. These have a combining weight of 306 to 307, and a specific gravity of 950 to 951 at 60° F. They are thick, viscid and of an oily appearance, and besides containing ricinoleic acid contain palmitic acid.

It takes from 17.5 to 18 per cent. of caustic potash or from 12.5 to 13.3 per cent. of caustic soda to saponify it, these quantities being rather less than is usual with the oils.

Castor oil is used with some degree of success in lubricating very heavy and quick running bearings of machinery: in such there is usually great friction accompanied by some heating, and castor oil resists those influences better than most other oils.

Castor oil cannot be mixed with hydrocarbon or mineral oils; it can be mixed with other fatty oils.

#### CONSTANTS OF CASTOR OIL.

Specific Gravity at 15° C. (60° F.) . . . . .	0.960 to 0.966.
"    "    "    100° C. (212° F.). . . . .	0.9096.
Solidifying Point, 17 to 18° C. (1° to 1° F.).	
Reichert Value, 1.6 cc. $\frac{N}{10}$ KOH.	
Saponification Value (Koettstorfer Test), 17.8 to 18 per cent. KOH.	
Iodine Value (Hubl Test), 83.6 to 84 per cent.	
Acetyl Value, 153.4 per cent.	
Maumene Test, 46° C.	
Viscosity at 70° F. . . . .	1220.
"    "    100° F. . . . .	270.
"    "    120° F. . . . .	170.
"    "    150° F. . . . .	129.

## CONSTANTS OF FATTY ACIDS OF CASTOR OIL.

Specific Gravity at 15° C. (60° F.)	. . .	0.9509.
" " " 100° C. (212° F.)	. . .	0.896.
Solidifying Point, 3° C. (27.5° F.).		
Melting Point, 13° C. (57° F.).		
Molecular Weight (Combining Equivalent),		292.
Iodine Value (Hubl Test),		90 per cent.

## OLIVE OIL.

The olive is the fruit of the tree *Olea Europea*, which grows very abundantly in those countries of Europe, Asia, and Africa that border on the Mediterranean. It is extensively cultivated in Italy, North Africa, Grecian Archipelago, Spain, and Asia Minor, from all which places olive oil is exported. The olive is a fruit resembling the plum, and of about the same size. There are certain variations of the olive grown in different localities due to climatical differences and in the mode of cultivation. The fruit is collected when just ripe, and in that condition it yields the finest quality of oil. Olive oil is yielded by the pericarp or pulp which surrounds the kernel. The kernel also is capable of yielding oil, but it is interesting to note that the oil yielded by the kernel is different to that given by the pulp. The olive oil is obtained from the fruit by pressure; some portion is also separated by use of solvents. The simplest method which has been in use for a long time consists in pressing in a primitive mortar, and separating the oil which flows out. Generally the pulp is put into a large tub or tank and subjected to pressure. The oil which flows out is known as "virgin oil". It is of fine quality, and used chiefly for edible purposes. There is a considerable proportion of oil left in the pulp, and this is usually extracted by boiling the pulp with water, then subjecting the residual pulp to a second pressure. A rather poorer quality of oil is thereby obtained. This quality of oil is chiefly used for lubricating, soapmaking, and general industries. A poorer quality is

often got by subjecting the residual cake from this second pressing to extraction by means of bisulphide of carbon. This gives a lower grade of oil used for the commonest purposes, and generally known as "sulphur" olive oil.

One of the troublesome processes in the making of olive oil is the separation of the oil from the watery juice after it comes from the oil press. The universal custom is to collect this mixture of water and oil as it drips from the press and leave it for several hours, then to skim off the oil that has risen to the top by reason of its lower specific gravity. This skimming must be repeated every few hours till the oil is entirely separated, for if not at once removed it begins to acquire a bad taste from the fermentable substances contained in the water. Besides the necessary labour, this process requires a large room and a very expensive outfit of large tanks. In order to avoid all this expense and trouble an apparatus has been made that performs the work automatically and continuously, enabling the oil maker to have clear, clean oil within two minutes from the time it leaves the press. The apparatus, shown in Figure 48, consists essentially of a tin tank about four feet high by two in diameter. This tank is kept constantly full of fresh water by means of a pipe connected with some adequate supply, the level being regulated by means of stopcock outlets. The juices and oil from the oil press, charged with oil in an emulsified state, are made to flow into the tank near the bottom, through a small "drum" perforated on the top, from which a stream of fresh water escapes in vertical jets. These two currents of oil and fresh water at once mix, and the oil passes upwards by reason of its lightness. Being in very small drops, it is washed of its heavier impurities (tissue, colouring matter, etc.) and reaches the top of the column of water in an almost perfect clean state, having left all grosser impurities to be carried off through an escape pipe at the bottom. When

sufficient oil has been collected at the top, a stopcock is opened, and the oil runs off, ready to be clarified.

Olive oil varies considerably in its quality. The best oils have a yellowish colour, while some of the inferior qualities are of a greenish-brown tint. In some cases the oil has a greenish tint. The specific gravity ranges from 0.916 to

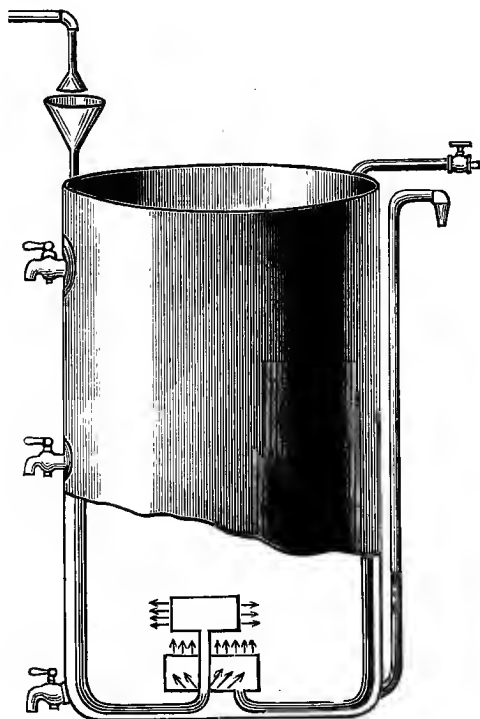


FIG. 48. Oil Separator.

0.919 at 60° F., the presence of much free acid lowering it. The best quality of olive oil contains usually about 2 per cent. of free acid. Certain grades of what are known as "huiles tournants," prepared from very ripe and fermented fruits, which are largely used in dyeing, contain as much as

25 per cent. of free acid. The odour of olive oil is pleasant and peculiar; the taste is sweet and bland. When cooled down olive oil deposits stearin and becomes solid at 6° C. (23° F.). It requires from 19.1 to 19.06 per cent. of KOH to saponify it. It absorbs iodine, and when mixed with sulphuric acid gives rise to an increase in temperature of 41° to 45° C. One property of olive oil is that, when mixed with nitrous acid or nitrate of mercury, it becomes solidified, being converted into elaidin. This property is not possessed to the same degree by any other oil. Olive oil is largely adulterated, the usual adulterants being cotton seed oil and mineral oils, but the character of the adulteration varies from time to time. The presence of cotton seed oil tends to increase the specific gravity, that of mineral oils tends to reduce it, while at the same time their addition reduces the flashing point of the oil. To determine the purity of olive oil regard must be paid to the specific gravity, flashing point, Koettstorfer test, Hubl iodine value, and the Maumene sulphuric acid test and elaidin test.

Olive oil is not as largely used now for lubricating machinery as formerly. It is often added to mineral oils in preparing spindle and loom oils. When free from acid it is a very good lubricant and works well. Olive oils which contain a large proportion of free acid are not suitable for lubricating machinery, the free acid having too strong an action on the machinery, forming soaps which cake on the bearings, and increase vibration and friction.

#### CONSTANTS OF OLIVE OIL.

Specific Gravity at 15° C. (60° F.)	. . .	0.916 to 0.919.
" " " 100° C. (212° F.)	. . .	0.862.
Solidifying Point, 6° C. (23° F.)		
Insoluble Fatty Acids (Hehner Value),		95.4 per cent.
Reichert Value, 0.3 cc. $\frac{N}{10}$ KOH.		
Saponification Value (Koettstorfer Test),		19.1 to 19.6 per cent. KOH.

Iodine Value, 80 to 83 per cent.	
Maumene Test, 41.5° to 45° C.	
Viscosity at 70° F. . . . .	120.
"    " 100° F. . . . .	60.
"    " 120° F. . . . .	45.
"    " 150° F. . . . .	30.

#### CONSTANTS OF FATTY ACIDS FROM OLIVE OIL.

Specific Gravity at 100° C. (212° F.), 0.8749.
Solidifying Point, 21° C. (70° F.).
Melting Point, 26° C. (79° F.).
Molecular Weight (Combining Equivalent), 280.
Iodine Value, 86 to 88 per cent.

#### RAPE AND COLZA OIL.

The oils known under these names are obtained from various species of *Brassica*, the rape and the cole plants, which are largely cultivated, especially in North Germany, France, Belgium, Russia, and India. The largest proportion of rape seed imported into this country comes from the Black Sea and Baltic ports. The oil is obtained from the seed by pressure, the yield of oil being from 30 to 45 per cent.; the crude oil being known as "brown rape oil," and containing a good quantity of moisture, mucilage, and colouring matter. This is refined into rape and colza oils. In this country colza oil is the name given to the most highly refined variety of rape oils. On the Continent it is the custom to distinguish the oils obtained from different species of the plant. Rape oil is refined by treatment first with sulphuric acid and then caustic soda. If colza oil is wanted these operations are repeated.

Refined rape oil has a pale yellow colour, is limpid, has a peculiar and characteristic odour, and an unpleasant harsh taste. When exposed to the air it becomes slightly more viscid. When boiled with caustic potash it yields a reddish colour, and requires 17.5 to 17.9 per cent. of caustic potash (KOH) to saponify it. Its saponification value is

about 320. It combines with a large proportion of iodine. Sulphuric acid has a strong action, giving an increase in temperature of from 64° to 65° C.; the specific gravity at 15° C. (60° F.) ranges from 0.913 to 0.915, at 100° C. (212° F.) the specific gravity is 0.863. Commercial samples of rape oil often contain small quantities of free fatty acids; the proportion ranges from 0.5 to 5.5 per cent. The acids found in rape oil consist chiefly of oleic and stearic acids; in addition there is rapic acid, and probably an acid of the linolenic series.

Rape oil has a considerable viscosity and hence has been largely used for lubricating machinery, but of late years mineral oils have taken its place. It is largely used in admixture with mineral oils for lubricating looms and other machinery. Its lubricating powers are very good. It has however a slight tendency to dry on the bearings or "gum"; the freer the oil from acid the less this tendency.

Rape oil is rarely adulterated, but when this is the case the fact of adulteration is readily ascertained.

#### CONSTANTS OF RAPE OIL.

Specific Gravity at 15° C. (60° F.)	. . .	0.913 to 0.916.
" " " 100° C. (212° F.)	. . .	0.8632.
Solidifying Point, 2° to 10° C. (30° to 14° F.)		
Insoluble Fatty Acids (Hehner Value), % per cent.		
Saponification Value (Koettstorfer Test),		17.5 to 17.9 per cent. KOH.
Iodine (Hubl Test),		100.8 to 102.6 per cent.
Reichert Test, 0.25 cc. $\frac{N}{IO}$ KOH.		
Maumene Test, 51° to 60° C.		
Viscosity at 70° F.	. . . . .	135.
" " 100° F.	. . . . .	55.
" " 120° F.	. . . . .	45.
" " 150° F.	. . . . .	28.

#### CONSTANTS OF FATTY ACIDS OF RAPE OIL.

Specific Gravity at 100° C. (212° F.),	0.8438.
Solidifying Point, 18.5° C. (65° F.).	
Melting Point, 18.5° to 21.5° C. (65° to 70° F.).	
Molecular Weight (Combining Weight),	320.
Iodine Value (Hubl Test),	99 to 103 per cent.

## GROUND NUT OR ARACHIS OIL.

This oil is obtained from the nuts of the *Arachis Hypogea*, cultivated in various countries (chiefly in Africa) on account of its oil-yielding qualities, the seeds containing some 45 to 50 per cent. of oil. Ground nut oil is of a pale yellow to almost colourless oil. It has a peculiar nutty odour and taste; its specific gravity ranges from 0.915 in the best qualities to 0.920 in the commoner qualities. It saponifies readily with caustic potash, taking from 19 to 19.5 per cent. of caustic potash (KOH) for complete saponification. It has but little tendency to become rancid on keeping, but when exposed in thin layers to the air it is rather more prone to oxidation than is olive and lard oils; hence it does not form a satisfactory lubricating oil, and although from time to time it is used for this purpose, yet it is but rarely employed.

In its chemical composition ground nut oil is somewhat peculiar, inasmuch as it contains palmitin, olein, stearin, and the glycerides of arachidic and hypogæic acids, which are almost peculiar to this oil. In many of its properties ground nut oil closely resembles olive oil. The presence and amount of arachis oil in any sample of oil, may be detected by separating out the arachidic acid. The best process for doing so is that devised by Renard. It is as follows: A quantity of the ground nut oil or other oil is thoroughly saponified by means of caustic potash or caustic soda, and the resulting soap decomposed by means of acids, the fatty acids being collected, well washed, and dried. 9.5 grammes of these acids (which may be considered as equal to 10 grammes of the original oil) are taken, dissolved in alcohol and a solution of lead acetate in alcohol added, which precipitates the fatty acids as lead soaps. These are collected on a filter, washed with a little alcohol, and then treated with ether several times





and linseed oil in its drying properties. It is therefore not suitable for lubricating machinery on account of its tendency to gum, owing to its oxidation by absorption of oxygen. It has been occasionally used for this purpose.

#### FISH OILS.

##### ARCTIC SPERM OIL.

This oil is obtained from the Arctic sperm or bottlenose whale (*Balaena Rostrata*). The oil is found in the blubber surrounding the body of the whale, and is extracted from it in the usual manner. As first obtained, the oil contains much spermaceti; this is extracted by refrigerating or cooling, and subjecting the cooled oil to pressure and filtering.

Arctic sperm oil is a limpid oil, of a pale yellow colour; its odour is, as a rule, in well-prepared oils but slight, and is of a fishy nature. Well-made sperm oil will deposit but little solid matter when subjected to cold.

It is not liable to become rancid by keeping. It retains its viscosity under the influence of heat better than any other oil. For lubricating quickly moving light machinery, like spinning spindles, there is no better oil, but it is not suitable for heavy machinery. It has no tendency to dry up and become gummy on the bearings, nor has it any corrosive action on them.

A very similar oil is the

##### SOUTHERN SPERM OIL.

This is obtained from the true sperm or cachalot whale (*Physeter Macrocephalus*), which is notable for the enormous size of the head. It is an inhabitant of the Pacific and Indian Oceans, but is becoming scarcer year by year. The Southern sperm oil is the original sperm oil, the Arctic variety being of more modern introduction.

This oil is found in the head cavity, a large vessel which will hold as much as 200 barrels full of oil. It is also obtained from the blubber of the whale. That from the head is usually considered to be of better quality than that from the blubber, but that is doubtful. On cooling, the oil as it comes from the whale deposits spermaceti, and the modern methods of refining have for their object the extraction of as much spermaceti as possible from the oil.

There does not seem to be any material difference between the two varieties of sperm oil except in price, the Southern sperm being the more expensive; some users consider it to lubricate better than does Arctic sperm. It will be convenient, therefore, to discuss the chemical and physical properties of these two oils together under the name of sperm oil.

Sperm oil is a thin, limpid oil of a pale yellow colour, having a fishy odour and taste which is but slight. It is very light in specific gravity—0·880 to 0·884 at 60° F.—if anything, Arctic being a fraction heavier than the Southern sperm oil. It is therefore the lightest of all natural oils. It differs markedly from all other oils inasmuch as it is not a glyceride, but resembles the waxes in its chemical composition, being a compound of fatty acids with alcohol radicles. The acid or acids belong to the oleic series, for it or they can be elaidinised, but up to the present they have not been isolated. The alcohols, also, have not been separated. The amount of caustic potash required to saponify sperm oils is low, from 12·3 to 14·7 per cent. KOH. The oil contains from 39 to 41 per cent. of alcoholic bodies. Sulphuric acid gives rise to an increase of heat of 47° to 51° C., and gives a yellowish brown mass, which distinguishes sperm oil from other fish oils. This oil absorbs from 81 to 84 per cent. of iodine. The two varieties of sperm oil cannot be distinguished from one another by any chemical test;

there are some small differences in the odour and taste by means of which an expert can distinguish between them.

Sperm oils are frequently adulterated, but their peculiar properties enable the analyst to detect this adulteration. The best tests to apply are the specific gravity, Koettstorfer test, and flash point. When boiled with caustic soda or potash, sperm oils give a red coloration.

#### CONSTANTS OF SPERM OIL.

Specific Gravity at 15° C. (60° F.)	. . .	0.875 to 0.883.
"    "    "    "    "    "    "    "    "    "    "	. . .	0.803.
Saponification Value (Koettstorfer Test),		12.5 to 14 per cent. KOH.
Iodine Value (Hubl Test),		84 per cent.
Reichert Value, 1.3 cc. $\frac{N}{10}$ KOH.		
Fatty Acids,		60 to 64 per cent.
Alcohols,		39 to 41 per cent.
Maumene Test, 51° C. (124° F.).		
Viscosity at 70° F.	. . . .	Arctic. 65 Southern. 63
"    "    "    "    "    "    "    "    "    "    "	. . . .	37      31
"    "    "    "    "    "    "    "    "    "    "	. . . .	29      26
"    "    "    "    "    "    "    "    "    "    "	. . . .	21      20

#### CONSTANTS OF FATTY ACIDS FROM SPERM OIL.

Specific Gravity at 15° C. (60° F.),	0.899.
Solidifying Point, 11° to 12° C. (52° to 53° F.).	
Melting Point, 13° C. (55° F.).	
Combining Equivalent (Molecular Weight),	281 to 290.
Iodine Value,	85 per cent.

#### WHALE OIL.

This is also known as train oil and sometimes as blubber oil. It is obtained from the blubber of various species of whales, of which the principal is the Greenland or right whale (*Balaena Mysticetus*), which is an inhabitant of the Arctic seas. From the blubber of this whale about 125 barrels of oil are obtained. The Polar whale (*Balaena Glacialis*) is found on the coasts of Greenland, Iceland and Northern Norway. The yield of oil from this whale aver-

ages about ninety barrels. The humpback whale (*Balænoptera Boops*) and the "finner" whale (*Balænoptera Gibbar*) are natives of the Northern seas; they yield a good quantity of oil, usually of good quality. The common whale (*Balæna Rostrata*) is a usual inhabitant of the seas north of Scotland, and yields a small amount of oil. Other species of whale are caught in the Southern seas. Generally, no attempt is made at keeping the oil from each species of whale distinct, although it is reasonable to suppose that there will be some difference in the chemical composition and properties of the oil obtained therefrom. The blubber of the whale varies in thickness from eight to twenty inches. At one time the oil was obtained from it by very crude means: the blubber was cut up into small pieces, placed on racks, and the oil allowed to drain from it. This it was enabled to do by the openness of texture of the blubber, and also by the animal tissue becoming decomposed. The result, however, was the production of a quality of oil, dark in colour, possessing a very strong odour and containing a great deal of free acid. To purify the oil it was usual to heat it up to a temperature of about 220° F., which destroyed the odour, and then to boil it with water for about an hour, after which it was allowed to settle and the oil run off into casks.

A better method is, however, now largely adopted. The fresh blubber is cut up into pieces and boiled with water to liberate the oil from it. This method is described in detail on page 132. This process yields an oil much paler in colour, freer from odour, and more neutral in properties than is obtained from the old method.

Whale oil is a very variable product. In colour it ranges from a straw yellow—"pale Norwegian whale oil"—to a reddish brown—"brown whale oil"—the fresher the blubber the paler the oil. Some varieties of crude whale oil deposit

stearin on cooling; this stearin is usually separated out and used in the preparation of soap. The specific gravity ranges from 0.920 to 0.931 at 60° F.; it requires from 19 to 20 per cent. of caustic potash to saponify it; it absorbs 80 per cent. of iodine and 50 per cent. of bromine. The increase in temperature with sulphuric acid ranges from 85 to 91° C. Sulphuric acid also produces a purple colour with whale oil. The chemical composition is rather variable; some samples contain glycerides of low fatty acids, and when subjected to the Reichert test, the distillate takes from 5 to 12 cc. of decinormal alkali. The fatty acid most common is valeric acid.

When boiled with caustic soda or caustic potash, whale oils give a red coloured soap.

Whale oil is a cheap oil and is rarely adulterated, and then usually with mineral oil. It is employed in soap-making, in illuminating, and to a small extent the better qualities are used as lubricants. In this capacity they give fairly good results when used on shafting, machinery bearings, and mixed with mineral oil they work fairly well for looms and spindles of textile machinery; it is important, however, that none but the best qualities should be used for this purpose.

#### CONSTANTS OF WHALE OIL.

Specific Gravity at 15° C. (60° F.) . . . . .	0.925.
"    "    "    100° C. (212° F.) . . . . .	0.8725.
Solidifying Temperature, 2° C. (30° F.).	
Insoluble Fatty Acids (Hegner Value), 93.5 per cent.	
Reichert Value, 2 to 12 cc. $\frac{N}{10}$ KOH.	
Saponification Value (Koettstorfer Test), 18.8 to 19.4 per cent. KOH.	
Iodine Value, 110 per cent.	
Maumene Test, 91° C.	
Viscosity at 70° F. . . . .	112.
"    "    100° F. . . . .	55.
"    "    120° F. . . . .	36.
"    "    150° F. . . . .	30.

## SEAL OIL.

Various species of seals, such as *Phoca foetida*, the harp seal, *Phoca Greenlandica*, the hooded seal, *Crystophora Cristata*, *Phoca barbata*, yield a fairly large quantity of oil varying of course in amount in the different species of seals, the larger varieties from 20 to 25 gallons being usually the yield. The seal fishery is a very important one in and around the coasts of Newfoundland, Greenland, and North America, the seals being captured for the sake of their skins and oil. The oil is extracted from the blubber by the same process as whale oil. No attempt appears to be made to keep the oil from various species of seal separate.

Seal oil, like whale oil, varies considerably from a light straw-coloured oil, with a very slight fishy odour, to a brown, strongly odorous oil; the specific gravity ranges from 0.924 to 0.929; it requires 19 per cent. of caustic potash to saponify it; it absorbs 91 per cent. of iodine or 57 per cent. of bromine. It is used for a variety of purposes, soapmaking, illuminating, and, to a small extent, for lubricating machinery, its lubricating properties being similar to those of whale oil.

## CONSTANTS OF SEAL OIL.

Specific Gravity at 15° C. (60° F.) . . . . .	0.915 to 0.920.
"    "    "    100° C. (212° F.) . . . . .	0.8733.
Solidifying Point, 3° C. (26.5° F.).	
Insoluble Fatty Acids (Hehner Value), 94 per cent.	
Reichert Value, 0.2 cc. $\frac{N}{10}$ KOH.	
Iodine Value, 127 to 130 per cent.	
Maumene Test, 92° C.	
Viscosity at 70° F. . . . .	75.
"    "    100° F. . . . .	50.
"    "    120° F. . . . .	32.
"    "    150° F. . . . .	25.

## PREPARED OILS.

## BLOWN OR THICKENED OIL.

Many oils, such as rape, cotton seed, olive, ground nut, sperm, neatsfoot, etc., have the property of absorbing oxygen

and thereby becoming thick and viscid. This property is taken advantage of in the production of heavy viscid oils used in conjunction with mineral oil for the purpose of preparing lubricants for heavy machinery. The two oils which are most commonly used for this purpose are rape oil and cotton seed oil, on account of the fact that they

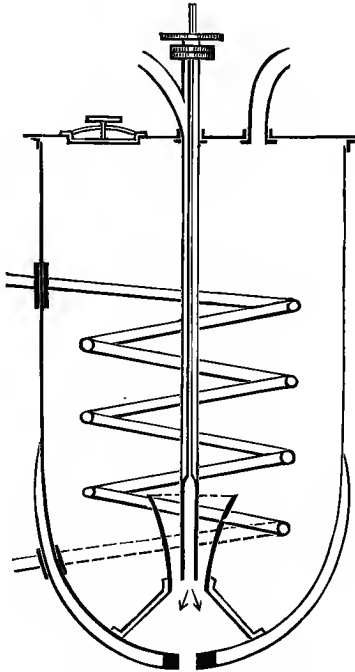


FIG. 49. Plant for Blowing Oils.

absorb oxygen much more quickly than the other oils, and can therefore be thickened up much quicker.

The operation may be carried out in a tall cylindrical pan, the bottom portion of which may be jacketed for the application of steam heat, or the steam may be sent through a steam coil. The arrangements ought to be made so that



a current of cold water can be passed either through the steam jacket or the steam coil for the purpose of regulating the temperature during the operation. Air is blown in through a vertical pipe which passes down nearly to the bottom, the air being blown in by an air pump, a rotary blower, or a centrifugal pump. For the purpose of bringing the oil into intimate contact with the air, the air pipe is usually made to terminate in a perforated coil or cross piece, or it may terminate in a perforated box.

Figure 49 is a drawing of Veitch Wilson's apparatus. The vertical air pipe is open at the bottom of the pan and about one inch above the opening in the bottom of the inverted truncated cone, the greatest diameter of which is about one-third of that of the pan, while the opening in the bottom is the same as that of the air pipe. The inverted cone is supported in the centre of the bottom of the pan by stays bolted by short screws to the sides and bottom of the pan. When air is driven through the central pipe it impinges upon the oil at the narrow opening in the cone, and by setting it in motion induces a downward suction and a circulating motion affecting the whole contents of the pan. The apparatus shown in Figure 44 for bleaching fats by air may also be used for this purpose.

The operation of thickening oils is carried on as follows: The pan is filled half full of oil. Steam is then sent into the jacket or coil, and the temperature of the oil raised to 160° or 170° F. The air which is sent in should also be heated to the same temperature. The air is then blown in. In a short time the oil begins to oxidise, and the temperature shows signs of rising. When this happens the steam is turned off or otherwise regulated, so as to maintain a uniform temperature. Should the temperature show signs of rising too much, then means must be taken by using the current of cold water or otherwise of reducing it down to that

required. The operation extends from eighteen hours to forty-eight hours, the variation in length of time being brought about by the differences in the quality of the product which it is desired to make, and by the conditions under which the operation is carried on. Given sufficient length of time, products may be obtained of almost any required degree of specific gravity up to 0.985 or 0.999, and varying in consistency from medium to very viscid or even solid oils. The temperature of working also has some influence, the higher it is the quicker the action proceeds; the products produced under these conditions are usually darker in colour, and have a stronger odour than those produced at low temperatures.

Thickened oils have a peculiar characteristic odour by means of which they can be readily distinguished when mixed with other oils. Boiled with caustic alkalies they give a dark red coloration. They yield from 85 to 90 per cent. of insoluble fatty acids, which shows that the operation of blowing converts some of the original insoluble acids of the oil into soluble ones. Another feature is that these acids are not entirely soluble in petroleum spirit, which would show that the acids had been converted into hydroxy acids. The flashing point of the oil is lowered considerably. The viscosity is increased ten to twenty times at the ordinary temperature, but it is reduced rapidly by heat. They usually contain more free acid than the unthickened oils.

Although these oils have now been in use for many years as lubricants without any complaints having been made, yet the author regards their use with some degree of suspicion as likely to lead to gumming and the formation of deposits on the machinery.

#### THICKENED RAPE OIL.

This oil is prepared from the ordinary rape oil; the usual specific gravity of the commercial article is 0.965 to 0.967,

very nearly that of castor oil. It is viscid in character, clear and transparent in appearance, light yellow in colour, and has a faint peculiar odour. It mixes well with all other oils, and is largely used for mixing with mineral oils. For the production of engine oils and of oils for heavy machinery it has been found fairly successful. The proportion usually employed is 10 to 20 per cent. of the thickened rape oil to 90 or 80 per cent. of mineral oil; in some cases other fat oils, such as neatsfoot or animal oil, are used in addition. A few figures relating to thickened rape oil are given below in the table of constants.

## CONSTANTS OF THICKENED RAPE OIL.

Specific Gravity at 15° C. (60° F.) . . . . .	0.9668.
"    "    "    "    100° C. (212° F.) . . . . .	0.9175.
Saponification Value (Koettstorfer Test), 22.12 per cent. KOH.	
Fatty Acids (Hehner Test), 87.5 per cent.	
Maumene Test, 65° C.	
Iodine Value (Hubl Test), 95.5 per cent.	
Bromine Value (Hehner Test), 63.5 per cent.	
Viscosity at 70° F. . . . .	1620.
"    "    100° F. . . . .	360.
"    "    120° F. . . . .	280.
"    "    150° F. . . . .	140.
Reichert Test, 1.2 cc. $\frac{N}{10}$ KOH.	
Flash Point . . . . .	360° F.
Fire Test . . . . .	520° F.

## LARDINE OIL.

This oil is prepared from cotton seed oil. It is made of various gravities ranging from 0.967 to 0.980. In most of its features it resembles thickened rape oil. It is usually, however, rather more cloudy in appearance, somewhat darker in colour, and has a stronger odour. It is used in the same way as thickened rape oil, and for the same purposes. The heavier grades of lardine do not mix thoroughly with mineral oils, and to enable them to blend it is needful to have a large proportion, 40 to 50 per cent., of the lardine,

or to use it in conjunction with some other fatty oil. The lighter grades of lardine oils mix fairly well with mineral oils in all proportions. The table below gives a few figures relating to this product.

## CONSTANTS OF LARDINE.

Specific Gravity at 15° C. (60° F.)	. . . . .	0.9817.
"    "    "    100° C. (212° F.)	. . . . .	0.9240.
Saponification Value (Koettstorfer Test), 24.64 per cent. KOH.		
Fatty Acids (Hegner Value), 91.4 per cent.		
Maumene Test, 75° C.		
Reichert Test, 1.3 cc. $\frac{N}{10}$ KOH.		
Viscosity at 70° F.	. . . . .	3010.
"    "    100° F.	. . . . .	1570.
"    "    120° F.	. . . . .	480.
"    "    150° F.	. . . . .	340.
Bromine Value (Hegner Test), 85 per cent.		
Iodine Value (Hubl Test), 128 per cent.		
Flash Point	. . . . .	400° F.
Fire Test	. . . . .	500° F.

## CHAPTER VI.

### TESTING AND ADULTERATION OF OILS.

It is not intended in this chapter to enter very fully into all the various tests that have been described and applied in the testing and analysis of oils, but simply to describe a few of the more simple and characteristic tests, so that oil dealers may be able to ascertain whether a sample of oil be pure, or whether it be a fit lubricant for the particular purpose for which it is to be used.

The practical analysis of oils is one surrounded by many difficulties, and to make a satisfactory analysis and to report on the purity of a suspected sample demands a large and varied experience among oils, an experience which it cannot be expected that an oil user should possess. It is therefore advisable to submit a suspected sample to a competent analyst, and it is desirable that such an analyst should have a special knowledge of oils. In fact it would pay large users of oil to make special terms with such analyst to test every lot of oil they have delivered to them, to see that it is what it is represented to be and that it is fit to use. In sending a sample to an analyst to be tested, from 6 to 8 ounces should be supplied, as satisfactory tests cannot be made with less.

In judging the purity or otherwise of a sample of oil the following tests are made :—

- 1st. Specific gravity.
- 2nd. Alkali tests.

- 3rd. Sulphuric acid tests.
- 4th. Free acid test.
- 5th. Viscosity test.
- 6th. Flashing point test.
- 7th. Evaporation test.
- 8th. Iodine test.

The 5th, 6th, and 7th are applicable solely to mineral oils or to mixed oils. It is useless to apply them to fatty oils, because with these oils these factors are fixed quantities and cannot be altered by any means. The method of making these various tests will be described, and then a few special tests for certain of the oils will be noticed.



FIG. 50. Specific Gravity Bottle.

1. *Specific gravity test.* There are three ways of determining the specific gravity of a sample of oil: 1st, by a specific gravity bottle; 2nd, by a hydrometer; 3rd, by the Westphal specific gravity balance.

The bottle method is the most accurate. The specific gravity bottle is one made specially for the purpose; it consists (Figure 50) of a small thin glass bottle, accurately stoppered, and the stopper has a small thin tube bored through it so that it will always hold the same volume of liquid when filled.

The bottle is carefully filled with the oil to be tested, taking care to avoid the formation of air bubbles, the stopper is carefully inserted, the outside wiped clean and dry, and the whole is weighed. The weight of water the bottle holds is also ascertained. Then the weight of the oil is divided by the weight of the water the bottle holds, and the result is the specific gravity of the oil: thus a sample of olive oil gave the following figures:—

Weight of bottle full of water . . . . .	38.496 grammes.
Weight of bottle . . . . .	13.496 ,,
Weight of water . . . . .	25.000 ,,
Weight of bottle full of oil . . . . .	36.387 ,,
Weight of bottle . . . . .	13.496 ,,
	22.891 ,,

$$\frac{22.891}{25} = 0.91564, \text{ specific gravity of the oil.}$$

Before filling the bottle with the oil, see that the temperature of the latter is 60° F., which is the standard temperature to which the specific gravities of all oils are referred.

Sprengel's tube, which is a U-shaped tube with capillary tubes (Figure 51), turned at right angles at the ends, is a very convenient piece of apparatus for the determination of specific gravities where only small quantities are available, or it is desired to find the gravity at a higher temperature

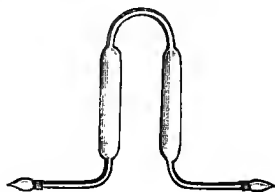


FIG. 51. Sprengel Gravity Tube.

than 60° F. The apparatus is used in the following manner : It is first weighed, then filled with water, and weighed again ; then filled with the oil whose gravity is to be tested, and weighed again. The tube, after being used for the water, should be cleaned out by first filling with methylated spirit, then with ether, and dried. If the gravity is to be determined at some higher temperature, it suffices to suspend the tube in a flask or beaker of water at that temperature, and keep it there for a short time, then take it out, dry the outside of the tube and weigh.

The hydrometer (Figure 52) method of determining specific gravity of oil is the most in use by oil dealers and consumers, and it is carried out as follows : The oil to be

tested is placed in a cylindrical glass jar (Figure 53) and the hydrometer is immersed in the oil. The degree on the scale of the latter instrument, which is level with the surface of the oil, is the specific gravity.



FIG. 52. Hydrometer.

Hydrometers are made provided with a variety of scales, but what the oil tester wants is two instruments, one with a scale ranging from 0.730 to 0.860, and the other with a scale from 0.860 to 1.000. The latter includes all the various grades of lubricating oils, and the former includes all the gravities of the lighter naphthas and burning oils. As a rule, the two mentioned will be found sufficient, but if greater accuracy is required then more instruments must be used to take the same range of gravities. The special form of hydrometer frequently sold under the name of "oleometer"



FIG. 53. Hydrometer Jar.

is perfectly useless and should never be used. Its divisions are arbitrary, and nobody seems to understand them. This method of determining the specific gravity is subject to many defects. A large quantity of oil is required to float the instrument properly, a quantity which is not always available. The accuracy of the instruments, as ordinarily sold, is not to be depended upon, inasmuch as the scale is not adjusted for each particular instrument, and it is impossible to make two alike in every respect—and to have one especially graduated to ensure accuracy is a costly proceeding—and there are other minor defects.

The Westphal balance (Figure 54) method is a more accurate one, and is just as easy to work as the hydrometer, while the results are perfectly reliable. The Westphal balance is simple and easy to understand, and gives the specific gravity directly. The principal part of the apparatus is a glass



bulb which has a volume of 5 cc. Consequently, when immersed in water, it requires the balance arm to which it is attached to be loaded with a weight of 5 grammes to restore the equilibrium. In liquids of lighter gravity proportionately a smaller weight would be required, or in heavier liquids a heavier weight. Riders of 5, 0·5, 0·05, and 0·005 grammes are supplied, and with these the

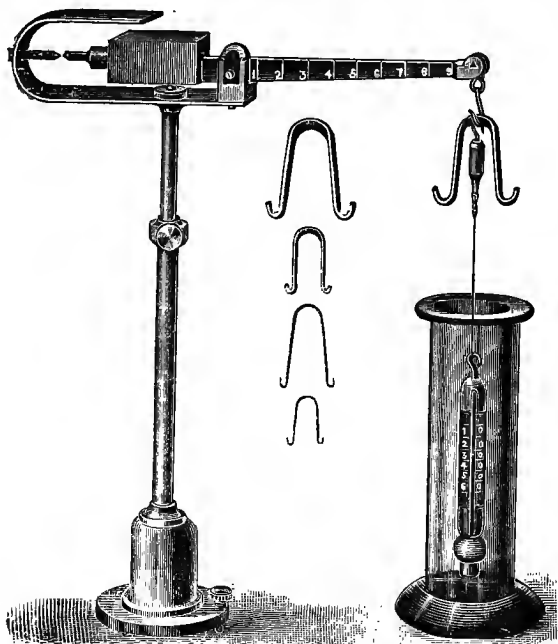


FIG. 54. Westphal Specific Gravity Balance.

specific gravity is easily ascertained. The position on the balance arm of these weights gives the gravity at once without any calculation being required. The balance is used as follows: It is mounted in position, and by means of the regulating screw at the base of the balance the two pointers are brought into line with one another. Then the glass jar is filled with oil, and the glass bulb immersed in

the latter. The large rider is now placed on the beam at the nearest division to produce equilibrium, then the next rider, and so on, until the two pointers are again brought into line. Then the numbers of the divisions are read off in the order of the weights, and these give the specific gravity. Thus, supposing the largest weight was on the 9, the next on the 1, the next on the 8, and the smallest on the 3 divisions of the balance arm, the specific gravity is 0.9183.

The glass plummet supplied with the instrument takes the form of a thermometer, the range of which is, however, only a few degrees on each side of 60° F. By substituting for this a plummet made of solid glass rod of exactly 5 cc. in volume, it is possible with the Westphal balance to obtain the specific gravity of oils at any temperature.

The balance does not give good results with very viscid liquids like glycerine, thickened rape and cotton seed oils, cylinder oils, and such should be tested with the bottle.

Temperature is an important element in testing the specific gravity of oils, and therefore the temperature of the oil at the time of testing must be noted. The standard temperature is 60° F. (15° C.), and, if possible, samples of oil should be brought to this temperature before testing. Temperature affects oils by decreasing the gravity as the temperature increases and *vice versa*. Although there are slight differences among the various oils as to the amount of variation brought about by temperature, yet these variations are slight, and, for the purposes of correction, may be neglected. The difference in the specific gravity of an oil for 1° F. is 0.00035, and for 1° C. 0.00063. Using these factors, correction for temperature may be made. Thus, suppose an oil has a specific gravity of 0.915 at 57° F. At 60° F., the standard temperature, it will have a gravity of  $0.915 - (0.00035 \times 3) = 0.91395$ . Similarly, an oil which at 64° F. has a specific gravity of 0.918, at 60° F. its gravity will be  $0.918 +$

$(0.00035 \times 4) = 0.9194$ . In other words, multiply 0.00035 by the number of degrees above or below 60° F. and add to or subtract from the specific gravity found according as the temperature is above or below the standard temperature of 60° F.

Too much stress must not be placed on the specific gravity test. Like most other oil tests, its indications are often of a negative character. It will not tell what an oil is, but what it is not. Thus it will not say that a given sample of sperm oil is pure, but it will say when an oil is not pure. Thus a sample of oil said to be sperm may have the right specific gravity (0.880), but for all that it may not be pure, and other tests must be made to decide this point. On the other hand, suppose that this test shows it to have a specific gravity of 0.870, the oil may be immediately condemned as impure.

The determination of the specific gravity of solid fats like tallow, palm oil, etc., is rather more troublesome than is that of liquid oils, and any method which may be adopted leaves room for doubt as to the accuracy of the result. A simple but only approximate method is to melt the fat at not too high a temperature. Then pour it into a specific gravity bottle and allow it to cool down to 15° C. (60° F.), and then weigh. This plan is objectionable on account of the great contraction which some fats undergo on cooling down, which may result in the bottle not being quite full of fat, and so a low and erroneous result will be obtained. Another plan is to have a wide glass tube fitted with a cover which can be pressed down tightly. The glass is first weighed full of water, then cleaned with the melted fat, which is allowed to cool down. The cover is put on and screwed down tightly, and the glass weighed again.

Another plan which is greatly used is to take advantage of the fact that fats or oils will just float in mixtures of

alcohol and water of the same specific gravity. The *modus operandi* is as follows: The fat is just melted and then allowed to drop into alcohol, whereby it is converted into spherical drops. Mixtures of alcohol and water of various gravities, 0.945, 0.940, 0.935, etc., are employed. The globules of fat are dropped in each of these alcohol solutions until one is found in which the fat floats; the specific gravity of that alcoholic solution corresponds with that of the fat.

TABLE OF SPECIFIC GRAVITIES OF FATTY OILS AT 15° C.  
(60° F.).

Almond Oil . . . . .	0.919
Arachis (Ground Nut) Oil . . . . .	0.920
Castor Oil . . . . .	0.964
Coconut Oil . . . . .	0.925
Cotton Seed Oil . . . . .	0.923
Linseed Oil . . . . .	0.932
Olive Oil . . . . .	0.915
Palm Oil . . . . .	0.940
Rape Oil . . . . .	0.914
Sesame Oil . . . . .	0.923
Lard Oil . . . . .	0.912
Tallow Oil . . . . .	0.912
Neatsfoot Oil . . . . .	0.914
Tallow . . . . .	0.940
Sperm Oil . . . . .	0.883
Whale Oil . . . . .	0.925

In the summaries of the constants given under each oil, gravities at other temperatures are given.

2. *Alkali tests.* As has already been explained the alkalis caustic soda and caustic potash convert the fatty oils into soap, but they have no action on hydrocarbon oils, except to form an emulsion from which the oil gradually separates out on standing.

Alkalies can be used in the testing of oils in three ways: first, to ascertain whether an oil is a pure fat or an hydrocarbon oil, or a mixture of both; and in the first case by noting differences in the colour and appearance

of the soap formed to determine the character of the fat oil present.

The method of applying this test is as follows: A solution of caustic soda or caustic potash (the latter has the strongest action on oils and very often gives the best results) is prepared, having a specific gravity of 1.340 (68° Tw.). Two volumes of this solution are shaken up in a test tube with four volumes of the oil. Fat oils will combine with the alkali and form an emulsion, from which very little oil will separate on standing, and the aqueous layer always has an emulsified appearance. Hydrocarbon oils only form a slight emulsion. The oil separates out on standing, leaving the aqueous layer quite clean or with only a faint cloudy appearance. A mixed oil will vary in appearance according to the proportion of the two oils present: if hydrocarbon oils are in the largest proportion, they will form a layer on the top and the aqueous layer will be emulsified; if the fat oil is in the largest proportion, then it will often be difficult to detect the mineral oil, but a little experience with this test will soon enable users of it to detect small quantities of hydrocarbon oil. A method of detecting mineral oils in fat oils which is more certain, and will show 2 or 3 per cent., is to dissolve a piece of caustic potash about the size of a pea in 5 cc. of alcohol, then add a few drops of the oil to be tested and boil for two or three minutes and add 3 or 4 cc. of distilled water. If the solution remains clear only a fatty oil is present. Mineral oil causes the solution to be turbid, and even so small a quantity as 2 per cent. will show itself in this way.

The amount of mineral oil in mixed oils is best ascertained as follows: 25 grammes of the oil are mixed with 10-15 cc. of the caustic alkali solution and 25 cc. of water and 5 cc. of alcohol; the mixture is boiled with constant stirring for about an hour, by that time the fat

oil will be saponified. The mixture is then put into a separating funnel, more warm water and 25 cc. of petroleum ether added. The whole is shaken together for a few minutes, then allowed to stand, when it separates into two layers. The upper layer consists of the petroleum ether with the mineral oil, the lower is an aqueous layer containing the soap made from the fatty oil. This is run off, clean water added, the mixture shaken up and again allowed to stand, and the aqueous layer again run off. This operation is repeated until the aqueous layer runs off clear. The ethereal layer is now run into a weighed glass, the ether evaporated off and the residual oil weighed. The weight multiplied by four gives the percentage of mineral oil in the sample.

Koettstorfer's saponification test is one of the most important tests that can be applied to oils, fat oils especially. This is carried out as follows: Two standard solutions are required: one of caustic potash dissolved in alcohol and containing about 28 grammes pure KOH in one litre of alcohol; the other is a solution of sulphuric acid containing 24.5 grammes  $H_2SO_4$  per litre. Both these are what are called by chemists semi-normal solutions. An alcohol solution of phenol phthalein is used as an indicator; this body is colourless, but alkalis turn it of a deep red colour; acids destroy this colour. The solution should be rendered of a faint pink tint by adding a drop or two of caustic potash.

Two grammes of the oil or fat are accurately weighed in a flask, and 25 cc. of the alcoholic solution of potash are measured and added. The flask has fitted to it a long glass tube which acts as a condenser. The flask with its condenser tube and contents is heated in a water bath, the flask being shaken at intervals till the oil is thoroughly saponified, which will take place in about thirty minutes. The flask is then removed, and the contents allowed to cool. A small

quantity of the phenol phthalein solution is added, and the standard acid solution run in from a burette drop by drop until the red colour of the mixture disappears. 25 cc. of the potash solution are now boiled in the flask (which has been previously cleaned out) alone for half an hour, and then the contents are titrated with the standard acid as before. The difference between the two amounts of acid used shows the quantity of potash required to saponify the oil. This difference multiplied by 0.028 gives the weight of KOH in grammes; this multiplied by 100 and divided by the weight of oil gives the weight of KOH (potassium hydroxide) required to saponify 100 parts of oil.

Methylated spirit may be used for making the alcohol solution of potash providing it be purified by distillation over lime and caustic soda as follows:—

The spirit is first placed in a bottle with a small quantity of quicklime and a piece of caustic soda, and allowed to stand for twenty-four hours, being shaken up at intervals. The spirit is next transferred to a retort or flask, and a little fresh lime and soda added. It is then distilled on the water bath till about 95 per cent. has come over; the remaining 5 per cent. is thrown away. This process frees the spirit from impurities which gradually act on the potash, cause it to become dark brown, and this coloration interferes somewhat with the operation of titrating with the acid. A slight brownness does not make much if any difference. It disappears along with the red colour due to the phenol phthalein at the end of the titration.

The reason for boiling 25 cc. of the potash alone is also due to the spirit or alcohol containing impurities which destroy the potash on boiling, and would therefore cause the oil to appear to have a high saponification value; but this error is eliminated by the method of carrying out the test described above.

As a rule, oils that resemble olive oil require from 19 to 19·5 per cent. of potash; rape oils from 17 to 17·6 per cent.; drying oils from 18·5 to 19 per cent.; whale oils, 18·75 per cent.; solid fats like tallow, 19·25 to 19·8 per cent.; coconut oil, 22 per cent.; butter, 24 per cent.; and sperm oils, 12·3 to 14 per cent. of potash for complete saponification.

Adulteration of fat oils with mineral oils would show itself by an abnormally low percentage of potash being required; and it would be possible to calculate the amount of adulteration from the figures which have been obtained by multiplying the percentage of potash by five, which will give approximately the percentage of fat oil in the sample.

3. *Sulphuric acid tests.* There are two different tests which can be made by means of sulphuric acid on oils. One is a colour test, the other is temperature test.

(1) *Colour test.* This is a most useful test for fat oils, but one where great experience and a close, observant eye are required. This test must be carried out only in bright daylight, so that shades of colour can be clearly discerned. It quite fails when done by gaslight or a dull daylight. The *modus operandi* can be varied somewhat, but it is essential in applying this test that it must always be made in the same manner, as the results are slightly different if the manner of making the test is varied. A good method is to place twenty drops of the oil in a clean white basin, and then add two drops of strong sulphuric acid. As the acid falls through the oil streaks of colour show themselves, and a tint of characteristic colour gradually spreads through the oil. After a minute or two the oil and acid can be stirred together, and the colour again noted. This test should first be made with samples of known purity, so as first to gain some experience of it, and, when testing unknown samples, comparative tests with pure oil should also be made. Vegetable oils give various colours, usually shades of yellow,



brown, or green ; fish oils turn off a violet or purple colour ; animal oils turn red or reddish brown. Hydrocarbon oils turn a blackish brown, but this effect is usually very slight.

The experimenter had best construct his own table of shades of colour. It is very difficult to convey by words what is actually meant by a colour name.

(2) *Temperature test.* This is known as Maumene's test, and is a very useful test and one that is easily made. 20 cubic centimetres of the oil are measured into a small beaker and a thermometer placed in it and its temperature noted. 8 cc. of strong sulphuric acid, sp. gr. 1.845, are then added and the oil and acid thoroughly stirred together with the thermometer as long as the temperature is observed to rise. The highest point which is reached is noted, and the initial temperature being subtracted the difference will be the increase in the temperature caused by the action of the acid on the oil ; and it will be found that the different oils show certain variations in the amount of increase they produce, as seen in the following table :—

Oils.	Increase in Temperature.	
	Degs. C.	Degs. F.
Olive Oil . . . . .	44	79.2
Sperm Oil . . . . .	37	66.6
Rape Oil . . . . .	35	63.0
Rape Oil (Thickened) . . . . .	58	104.4
Cotton Seed Oil . . . . .	65	117.0
Neatsfoot Oil . . . . .	32	57.6
Tallow Oil . . . . .	38	67.6
Castor Oil . . . . .	56	97.2
Rosin Oil . . . . .	25	45.0
Petroleum Lubricating Oil . . . . .	25	54.0
Scotch Shale Oil . . . . .	6	11.0
Linseed Oil . . . . .	68	122.4

These figures have been obtained by the writer when working with this test. It will be noticed that there is a marked difference between mineral oils and fat oils, the former giving much lower figures than the latter;

and between drying and non-drying oils, drying oils yielding much higher figures, the action is more energetic, and there is generally a quantity of sulphurous acid gas evolved (distinguished by its odour of burning sulphur); differences may also be noticed when carrying out this test on the colour and consistence of the resulting mass, which may be utilised as qualitative tests for the oils. The thickened rape and cotton oils are more heated than the normal oils. These figures must not be taken as a standard of comparison, but they are simply given to show the general tendency of this valuable test. It is found that different observers obtain slightly different figures, although their own figures are concordant enough. This is due to slight differences in the conditions of carrying out the test, which will naturally vary with each observer. Hence this test must be conducted in a comparative manner, samples of unknown purity with samples of known purity.

With this test it is possible to ascertain approximately the proportion of the constituents of a mixed oil when those constituents are known; thus, supposing an oil consists of a mixture of cotton seed and olive oils, it will yield an increase of temperature between  $44^{\circ}$  and  $65^{\circ}$  C., according to the relative proportions of the two. This is worked out by a formula:—

$$X = \frac{(C - B) \times 100}{A - B}$$

Where X = percentage of oil A in sample.

A = mean rise for pure sample of oil A.

B =           "           "           "           "           B.

C = observed rise in the mixed sample.

Thus, supposing in a mixture of olive oil and cotton seed oil the observed rise was  $56^{\circ}$  C., then according to the above formula we have

$$\frac{(56 - 44) \times 100}{65 - 44} = \frac{12 \times 100}{21} = 57.1 \text{ per cent. of oil A.}$$

Then there are 57.1 per cent. cotton seed oil and 42.9 per cent. olive oil in the mixed sample.

4. *Free acid test.* It is important that lubricating oils should be free from acid whether this be of fatty or mineral origin, as such free acid has a destructive effect on the metal of machinery, and it is astonishing what a corroding effect a small quantity of free acid in oil has on metals, especially on brass or copper when the two bodies have been in contact for some time. If an oil containing 3 per cent. of free acid be left in contact with brass for 12 hours it will have acquired a green tint, showing that it has dissolved some of the metal.

Fat oils such as olive, rape, castor, cotton, generally contain small quantities of free acid, rarely less than 1 per cent., and the writer has found as much as 22 per cent. of free fatty acid present in oils. This free acid may have been present originally in the oil owing to defective methods of extraction, or if the sample be an old one may have developed by keeping.

All fatty oils on keeping for some time slowly become rancid, some oils more rapidly than others. This rancidity is brought about by the combined action of the oxygen and moisture present in the air with which they are in contact, and results in the decomposition and splitting up of the oil into its two constituent parts—glycerine and fatty acid. Fatty acids have a strong corroding action on metal.

Mineral oils are usually free from acid. If any be present it is most likely to be sulphuric acid, and indicates imperfect washing of the oil during the process of manufacture.

A simple test for the detection of acidity in oils is the following: Make a solution of phenol phthalein in methylated spirit, as much of the former as will stand on a sixpence in about 6 ozs. of the spirit. Add to the solution a few drops of caustic soda solution until the liquor has acquired a perceptible red tint. Then take a little of the oil

to be tested, add a small quantity of the above test solution, and shake well. If there be any acid in the oil, the red colour of the test solution will be discharged. No other substance is capable of detecting traces of acid in oil. Litmus, which is much used by many persons, is of no use for this purpose.

The amount of free acid in oils may be readily determined thus: 10 grammes of the oil are weighed into a clean glass beaker, and 10 cc. of neutral methylated spirit or alcohol added with stirring, then 1 or 2 cc. of the phenol phthalein test solution are added. A standard decinormal solution of caustic potash or soda is then run in slowly from a burette, constantly stirring all the while, until a permanent red colour is obtained. As each drop of alkali solution falls into the oil it produces a pink spot. As long as any acid is in the oil this disappears on stirring. As soon, however, as the acid is neutralised the pink colour remains permanent. Each cc. of alkali solution used is equal to 0.0282 of free oleic acid. The result, multiplied by ten, gives the percentage of acid in the oil.

If it is necessary to distinguish mineral acids from fat acids in oil, a solution of methyl orange may be used. This is turned pink by mineral acids, such as sulphuric acid, but is not affected by fatty acids.

5. *Viscosity test.* The viscosity of lubricating oil is one of its most important properties, and therefore the method of determining this must necessarily occupy a prominent position in testing oils.

Viscosity or body of oils is a term used to indicate, to some extent at least, the relative fluidity of oils. Those which flow freely are said to be thin, "have no body," while those like castor oil which do not flow freely, or are viscid oils, are said to have "body". This viscosity of oils, as usually understood, is due to two properties of the oil, cohesion and adhesion, which exert a most important influence

on their value as lubricants. Cohesion binds the particles of the oil together. The greater this is the more viscosity the oil possesses and *vice versa*. Further, the more cohesion there is between the particles of oil, the greater pressure or force they will resist before splitting apart. It therefore follows that a viscid oil will lubricate better heavy machinery, where the pressure is great, than a thin oil, whose particles would be forced asunder under the pressure. Adhesion is another important function of oils comprised in viscosity. Adhesion is the term used to express the property of adhering to other bodies. The greater this is the better lubricant the oil must be, because, in virtue of it, it will stick or adhere closely to the surface of the bearings, and better resist the pressure brought to bear on them. A liquid may possess great cohesive but very little adhesive properties, as, for example, mercury. On the other hand, water, spirits, etc., have great adhesive force and but little cohesion, which makes them very limpid, easy-flowing liquids.

A good lubricant must possess both functions. It must be at once cohesive and adhesive, and the joint effect is expressed in the term viscosity, or body of the lubricant.

It is now generally accepted among oil dealers and oil consumers that the viscosity of an oil is a good measure of its lubricating value. Given two oils for lubricating, say, a spindle. The one having the most viscosity will have the most lubricating power, but, it has been pointed out before, it does not follow that a highly viscid oil like castor would lubricate a spindle better than a limpid oil like sperm. The viscosity of an oil must be adapted to the work it has to do, and this point should never be lost sight of either by oil consumers or oil dealers.

A common method of testing the viscosity of an oil is to fill a glass pipette with the oil, and note how many seconds it takes to run out. It is obvious that this method is rather unsatisfactory, as no account is taken of the temperature of the oil, which has an important influence in the viscosity of oils.

A better apparatus, and one very extensively employed, is that known as Sacher's, and which is shown in Figure 55. This consists of an inner glass tube of about 125 cubic centimetres capacity and graduated into 100 divisions of 1 cc. each

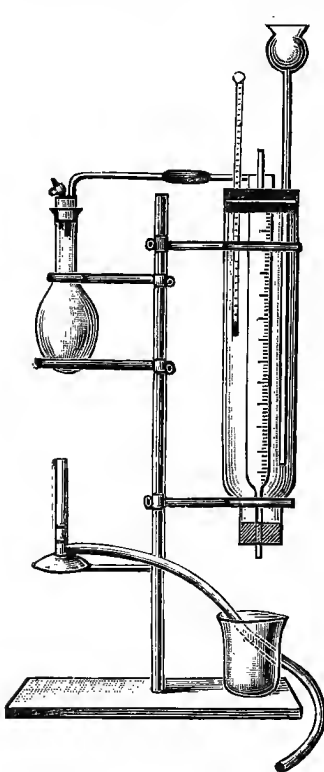


FIG. 55. Sacher's Viscosity Apparatus.

through part of its length. The lower part of this tube terminates in a short tube of narrow bore. This tube is surrounded by a wider glass tube which serves as a water jacket, and into this steam can be passed from a boiler so as to heat the water and the oil up to any required degree. The apparatus is used thus: The water is heated in the jacket to the required temperature at which the test is to be made, a thermometer being suspended in it for that purpose. The oil is heated in a separate beaker to a few degrees above the temperature at which the test is to be made. It is then poured into the inner tube, filling it up to the topmost division of the graduation. The oil is then allowed to flow

out, the time in seconds it takes for the 100 cc. to flow out being taken as the measure of the viscosity of the oil. The best temperatures for testing viscosity are 70° F., 100° F., 120° F., 180° F., and 212° F.

There are several objections to this apparatus: 1st. It is difficult to keep the temperature uniform throughout the test, and the oil is not at an equal temperature throughout

the whole of its volume. The top is always hotter than the bottom portions. 2nd. The oil does not run out at uniform speeds during the continuance of the test, the first portions running out much quicker than the last; hence it is necessary to always use the same volume of oil in all tests. This is not always possible, so many observers are in the habit of noting the time each 25 cc. takes to run out, and thus, if they have only about 25 cc. of a sample to work with, it is still possible to compare its viscosity with other samples. 3rd. A general standard of reference between different observers cannot be obtained with this instrument, as, being constructed of glass, it is impossible to make two which are identical in all respects, and give the same figures, which is an essential quality in viscometers. 4th. Being constructed of glass, it is fragile and very liable to break, and a breakage means a loss of any results obtained with the instrument, as new ones give different figures not comparable with those given with the old instrument.

The principal objections are the want of permanency and the non-standard character of the apparatus. The first objection can be got over by constructing the apparatus of metal; the last is by no means easy to overcome. Boverton Redwood's Standard Viscometer is constructed on a similar principle to the glass apparatus of Sacher's. There is an inner oil chamber, constructed of copper, silvered on the inside. This is surrounded by an outer vessel which holds water that can be heated very conveniently. The inner chamber has in the centre of its bottom a piece of agate, through which a fine aperture has been bored. This is so constructed that it can be closed by a ball valve. Being constructed of metal the apparatus can always be made of the same size, and the holes being of agate can also be always bored of the same size. In this way two instruments can be made which will give identical results. This is a most important point.

The instrument is used as follows: The inner oil chamber is filled with oil up to the gauge pin, thermometers are immersed in the oil and in the water, the latter is heated up to the temperature at which the test is to be made. This heats the oil in its turn. When the oil has attained the required temperature, the valve is lifted and the oil allowed to run out; the time taken to run out 50 cc. of oil being taken as the measure of the viscosity of the oil. The following are some figures given by Boverton Redwood (*Journal Soc. Chem. Ind.*, 1886, p. 128) as obtained with this instrument:—

VISCOSITY.—SECONDS FOR 50 cc.

Temperature. Fahr.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.
50	...	712 $\frac{1}{2}$	...	620	...	115	425	1030	2040	2520	...
60	25 $\frac{1}{2}$	540	177	470	...	105	295 $\frac{1}{2}$	680	1235	1980	...
70	...	405	136 $\frac{4}{5}$	366	...	90	225	485	820	1320	...
80	...	326	113	280	...	73	171	375	580	900	...
90	...	260	96	219 $\frac{1}{4}$	...	63 $\frac{1}{2}$	136	262	426	640	...
100	...	213 $\frac{1}{2}$	80 $\frac{1}{2}$	174 $\frac{3}{4}$	...	54	111	200	315	440	1015
110	...	169	70 $\frac{1}{2}$	147 $\frac{4}{5}$	...	50	89 $\frac{1}{2}$	153	226	335	739 $\frac{1}{2}$
120	...	147	60 $\frac{1}{2}$	126	...	47	78	126	174	245	531
130	...	123 $\frac{1}{2}$	57	112	...	44 $\frac{3}{4}$	63 $\frac{1}{2}$	101	135 $\frac{1}{2}$	185	398 $\frac{1}{2}$
140	...	105 $\frac{1}{2}$	50 $\frac{3}{4}$	88 $\frac{2}{5}$	...	41	58	82	116	145	317 $\frac{1}{2}$
150	...	95 $\frac{1}{2}$	49	75 $\frac{1}{2}$	...	37 $\frac{1}{2}$	52	70 $\frac{1}{2}$	95	115	250
160	...	85	47 $\frac{1}{2}$	70	...	...	46	63	83 $\frac{1}{2}$	93 $\frac{1}{2}$	200
170	...	76	46	62	...	...	...	58	70 $\frac{1}{2}$	77 $\frac{1}{2}$	161
180	...	69	44 $\frac{1}{2}$	56 $\frac{1}{2}$	...	...	...	52 $\frac{1}{2}$	61 $\frac{1}{2}$	67 $\frac{1}{2}$	134 $\frac{1}{2}$
190	...	64 $\frac{1}{2}$	43	53	...	...	...	47	56	61	115 $\frac{1}{2}$
200	...	58 $\frac{1}{2}$	42	50 $\frac{2}{5}$	54 $\frac{3}{4}$	...	...	42	48 $\frac{1}{2}$	54	99 $\frac{1}{4}$
210	...	54	40 $\frac{3}{4}$	48 $\frac{1}{2}$	...	...	...	40	...	...	85
220	...	50	39	47	...	...	...	38	...	...	77
230	...	47 $\frac{1}{4}$	36 $\frac{3}{4}$	45 $\frac{4}{5}$	...	...	...	...	...	...	70 $\frac{1}{2}$
240	...	45 $\frac{1}{2}$	35 $\frac{3}{4}$	44 $\frac{3}{5}$	...	...	...	...	...	...	64 $\frac{1}{2}$
250	...	43 $\frac{1}{2}$	34 $\frac{3}{4}$	44	...	...	...	...	...	...	59 $\frac{1}{4}$
260	...	...	33 $\frac{3}{4}$	43 $\frac{1}{2}$	40	...	...	...	...	...	54
270	...	...	32 $\frac{3}{4}$	43	...	...	...	...	...	...	48 $\frac{1}{2}$
280	...	...	31 $\frac{1}{4}$	41 $\frac{1}{2}$	...	...	...	...	...	...	46 $\frac{1}{2}$
290	...	...	30 $\frac{3}{4}$	41	...	...	...	...	...	...	44 $\frac{1}{4}$
300	...	...	30	38	...	...	...	...	...	...	42 $\frac{3}{4}$
310	...	...	...	35	...	...	...	...	...	...	...
320	...	...	...	33 $\frac{4}{5}$	...	...	...	...	...	...	...

- |                                  |   |       |
|----------------------------------|---|-------|
| 1. Water.                        | 6. American mineral oil, sp. gr.          | ·885. |
| 2. Refined rape oil.             | 7. " "                                    | ·913. |
| 3. Sperm oil.                    | 8. " "                                    | ·923. |
| 4. Neatsfoot oil.                | 9. " "                                    | ·909. |
| 5. Beef tallow.                  | 10. " "                                   | ·915. |
| 11. Russian mineral oil, sp. gr. | ·884 (semi-solid at common temperatures). |       |



Redwood recommends that the observed viscosity (rate of flow) be corrected for the effect of specific gravity on the flowing of the oil by multiplying the observed viscosity by the specific gravity of the oil, and dividing it by the specific gravity of the oil which has been selected as the standard.

The author considers this an unnecessary proceeding, and prefers to take the figures which are given by the viscometer as being correct, and to give the true viscosity of an oil.

One main objection to Boverton Redwood's instrument is the smallness of the aperture, which causes the time taken for the oils to flow out to be too long, thereby increasing the difficulty of keeping the temperature uniform throughout the duration of the test. There is another objection. It has been pointed out that the viscosity of an oil is due to a conjunction of two functions of the oil, cohesion and adhesion. Now when any oil flows out, as it does in Redwood's instrument, from a simple aperture in a metallic plate only, the function of cohesion is tested, while that of adhesion, which is most important, is not tested at all. In viscometers constructed on the flowing principle, the adhesion function of an oil can only be tested by causing it to flow through a narrow tube.

The author has devised a viscometer shown in Figure 56. This instrument has been devised to be a standard one, and not too costly. As will be seen from the drawing, it consists essentially of an inner oil chamber constructed of copper. This terminates in a short brass tube of narrow bore, the aperture of which is closed by a valve. Surrounding the inner oil chamber is a water-jacket, the water in which can be heated by a separate boiler working on the hot-water circulating system; a thermometer can be immersed in the water in the jacket and one in the oil. The inner oil chamber is filled up to a gauge pin with the oil, the water is

then heated to a few degrees—4 or 5—above the temperature at which the oils are to be tested, and it is maintained at this temperature during the test. This is easily accomplished. When the oil has attained the required temperature, the valve is opened and the oil allowed to flow out into a

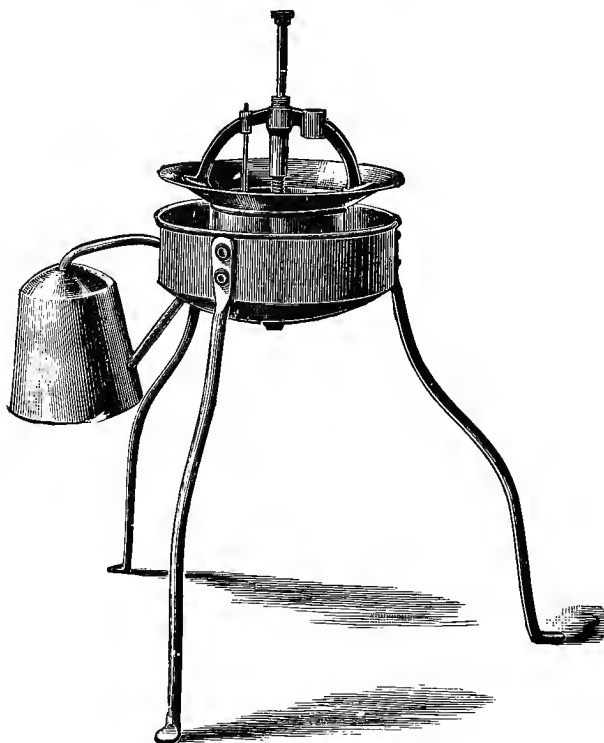


FIG. 56. Hurst's Viscometer.

measuring glass or flask, the time in seconds taken for 50 cc. to flow out being taken as the viscosity of the oil. The instrument is very easy to manipulate, and gives very concordant results. Some figures are given on p. 236.

Viscometers constructed on the principles of the one just described take no account of the varying specific gravities of

the different oils, and this must have some influence on the length of time that the oil will flow out, as heavy oil will, owing to its greater specific gravity, have a tendency to flow out quicker than a light oil whose specific gravity is less, because the pressure owing to gravity is greater in one case than the other.

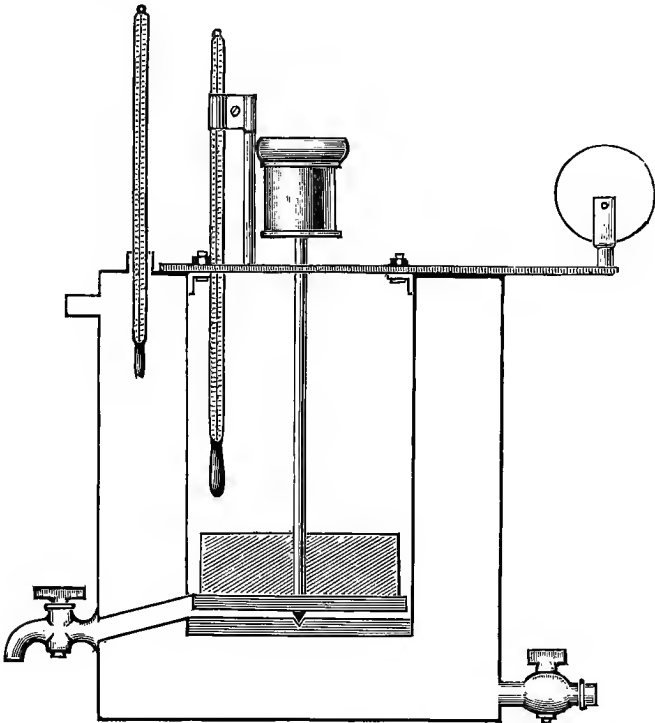


FIG. 57. Viscometer.

In the writer's viscometer this is eliminated as much as possible by the oil container being made short and wide, but it can never be entirely got rid of in this class of instruments. It is eliminated in another form of viscometer.

Mr. John Peters of Accrington has devised a viscometer

on another principle. In this the effect produced by gravity is got rid of. The apparatus is shown in Figure 57. It consists of an oil chamber, the bottom of which is made of a heavy disc of brass, the face of which is turned true in a lathe; a similar disc of brass is carried on a spindle and supported on the bottom piece on a pivot; the two brasses do not touch one another, but are carefully set a small distance apart. On the top of the upper brass is fixed a pair of fans forming a kind of paddle. The disc paddle is made to revolve by the falling of a weight attached to a string acting on a pulley or drum on the top of the spindle, and the relative viscosities of different oils materially affect the speed at which the paddle revolves, and therefore the time taken to fall a given distance is a measure of the viscosity of the oils. The time can be regulated to some extent, but it is usual to have a weight that will when rape oil is being tested run down about 3 feet in 100 seconds. This apparatus gives good results, and one instrument can be compared with another, so that it is quite standard.

Cottrell's viscometer resembles Peters', but it has not the faced brasses. Napier's instrument has a number of concentric rings, which revolve one in the other. Both these instruments are actuated by the falling of weights.

The main fault of these frictional viscometers, if they might be so called, seems to arise from the fact that their scope of action is not wide enough. If the weight is adapted to just pull the paddles round in a thick viscid oil like castor, it is too heavy for light hydrocarbon oils, say a '865 Scotch oil, and runs down too quickly in such oils, so that it is difficult to distinguish small differences in the viscosity of two samples of lubricating oil which closely resemble one another. This difficulty is experienced in a greater degree when

working at high temperature, when the viscosities of oils are very nearly alike. This difficulty may be overcome to some extent by using a set of weights, light for light oils and high temperatures, medium for medium oils, and a heavy weight for viscous oils, only there is a risk of not getting properly comparative results.

It is customary to take rape oil as a standard = 100, at 70° F., in stating the viscosity of an oil. On the paddle form of instrument the weight can be so adjusted that it will fall the given distance in 100 seconds and then the instrument will give the relative viscosity of other oils (compared with rape oil = 100) without further calculation.

Rape oil is taken as a standard because it is an oil with a good viscosity, and can be obtained without much trouble fairly pure and of a uniform quality.

With the flowing forms of viscometer a simple proportion sum will give the relative viscosities. Thus let A = the viscosity of rape oil, B the viscosity of the oil tested, then  $\frac{A \times 100}{B} = X$ , the relative viscosity of the oil. It has been proposed to eliminate the supposed effect of gravity on the rate of flow of the oil by multiplying the viscosity of the oil by its gravity, and dividing the result by the viscosity of rape oil, multiplied by its specific gravity. Such is called by some writers "specific viscosity". There is no advantage to be gained by the use of this new term, and as it is not very clear to most persons what it means, it is best to stick to the well understood method of stating viscosities.

The following table gives the relative viscosities of various oils at different temperatures :—

## VISCOSITIES OF OILS.

STANDARD RAPE OIL AT 70° F. = 100.

	70° F.	120° F.	180° F.	212° F.
Scotch Mineral Oils—				
865 sp. gr. . . . .	20	15	12	11
885 " . . . . .	45	16	13	12
890 " . . . . .	63	20	14	13·5
American Mineral Oils—				
885 sp. gr. . . . .	36	16	11	10·5
910 " . . . . .	85	25	12	12
915 " . . . . .	90	26	15	12·5
920 " . . . . .	127	30	20	17
Cylinder Oil . . . . .	1860	230	55	43
Summer Dark Machinery .	1300	90	33	21
Medium " . . . . .	245	50	20	17
Russian Mineral Oils—				
896 sp. gr. . . . .	66	26	16	12
908 " . . . . .	122	47	20	14
914 " . . . . .	316	94	24	17
910 " Dark . . . . .	236	78	22	16
Rosin Oil . . . . .	221	22	12	12

This table shows the influence of temperature on these oils. It will be noticed that they all become much more fluid as the temperature rises, and with few exceptions that there is not as much difference between them at the boiling point (10·5 to 17) as there is in the viscosities at 70° F. (20 to 1860). This is a curious feature. It will be noticed that the fluidity increases very rapidly between 120° F. and 180° F., and that there is very little difference between the viscosity of an oil at 180° to 212° F. The Russian oils it will be seen are, compared with their gravities, more viscid oils than the American oils, but they lose this much more rapidly by heat. The author is of opinion, as the result of much observation on the behaviour of oils in regard to their viscosity at different temperatures, that there is what may be called a "critical point," at which temperature the oil begins to lose its viscosity more rapidly, and below which point the loss of viscosity on heating is very slow; this

critical point varies with different oils, but the author has not been able to do more than make a few tentative experiments on the subject.

6. *Flash test.* This is one of the most important tests to apply to mixed mineral or lubricating oils. It is most easily carried out in the following way:—

Procure a white procelain basin of about 2 ozs. capacity, or better a copper vessel, and support it by suitable means over a bunsen burner, nearly fill it with the oil to be tested, and hang a thermometer in the oil. The bulb of the latter should be completely covered by the oil, while it should not be allowed to touch the bottom or sides of the basin. Connect with another gas supply an ordinary mouth blow-pipe, and adjust the supply of gas to this so that the flame at the jet is only about the size of a small pea. Then heat the oil, carefully adjusting the size of the bunsen flame so that the heating of the oil is not carried on too rapidly; a rise of 10° F. per minute is the generally recognised amount of heating, and it is not advisable to exceed this amount. A slow rate gives much more accurate results than a quick rate of heating.

During the progress of this test three points can be observed: 1st, the vaporising point; 2nd, the flash point; 3rd, the burning point or fire test.

The vaporising point. This is the first point to be observed. It is the temperature at which a perceptible vapour begins to be perceived. This is also known as the "smoke point". As a rule it is not considered of much importance, and what particular value it has is somewhat uncertain. Of course oils with a low vaporising temperature, say of less than 150° to 160° F., would be unpleasant to work with on machinery where that temperature is likely to be exceeded.

The method of carrying out and the rapidity of heating

have a considerable influence on the temperature at which an oil will begin to give off vapour. If the basin is heated rapidly over a naked flame the temperature of vaporising will be much less than when the basin is heated on a sand bath. A difference of 20° or even 30° F. may often be obtained by these two different methods of heating the oil. Those dealers and consumers who attach particular importance to the smoke point always immerse the basin of oil in a deep sand bath, taking care that it is well surrounded with sand. It is only by this means that different observers can obtain concordant results on the smoke point.

The flash point. If, while the oil is being heated, the small gas jet is applied at short intervals, say every two or three degrees of rise in the temperature, taking care not to let the flame touch the surface of the oil, soon a blue flame will be observed to shoot across the surface of the oil. The temperature at which this flame is first seen is the flash point.

Whether a sand bath or a naked flame is used for heating the oil makes but little difference in the flash point, rarely more than what different observers would note with the same oil. The higher the flash point the safer is the oil to use, because then there is no chance of it giving off any inflammable vapour at any temperature to which it may be subjected in actual use. This point of inflammability is of particular importance to cotton mills, where there is such a large quantity of fluff always about the machinery. If an oil with a low flash point were used and the temperature should by accident get above the flash point, and a light come in contact, it is quite possible that the oil would catch fire, and this would be sure to spread through the fluff and hence probably through the mill, so that it is of particular importance in a cotton mill to use an oil with a sufficiently high flash point. 350° F. is quite a safe point, and there is no



necessity to much exceed this point. Some dealers pride themselves on the high points of their oils, but a high flash point is not all that is wanted in an oil. There is the proper viscosity to be considered, and, generally speaking, high flash points and high viscosities go together, and thus where you want, as spindle oils, a moderate viscosity only, you cannot have very high flash points, and if the latter feature is only considered, then an oil may be used which, owing to its high viscosity, is not the most suitable for the purposes. For use in cotton and other textile mills, where the peculiar conditions and material introduce a special element of danger, the oils should not flash at a lower temperature than  $350^{\circ}$  F., while for all other mills and works  $330^{\circ}$  F. may be accepted as a safe point. For cylinder oils  $500^{\circ}$  F. is the lowest flashing point usually accepted as safe, but in the case of oils for lubricating cylinders there cannot be any question of safety because they are used under such circumstances as to preclude any idea of their taking fire. The true test of the value of a cylinder oil is its having a good viscosity which it does not lose to any great extent on being heated.

There is, however, a connection between a high flash point and a high viscosity in cylinder oils. The process of manufacture consists in eliminating all light portions, the presence of which would reduce the flash point and the viscosity considerably. The flash point is much easier to ascertain than the viscosity, hence the reason attention is given to it, not from any considerations of safety.

The burning point. This is known in America as the "fire test". If, after the flash point has been determined, the heating of the oil be continued, it will be observed that the flashing becomes more frequent and the flame gets more luminous. Presently the flame, instead of going out, will continue to burn. The temperature at which this occurs is "the burning point" or "fire test". This point is not



temperature indicated by the thermometer is noted as the flash point.

The flash point by the closed test is always lower, from 12 to 20 degrees, than by the open test.

The author does not regard the closed flash test as thus carried out with much favour. He finds that different

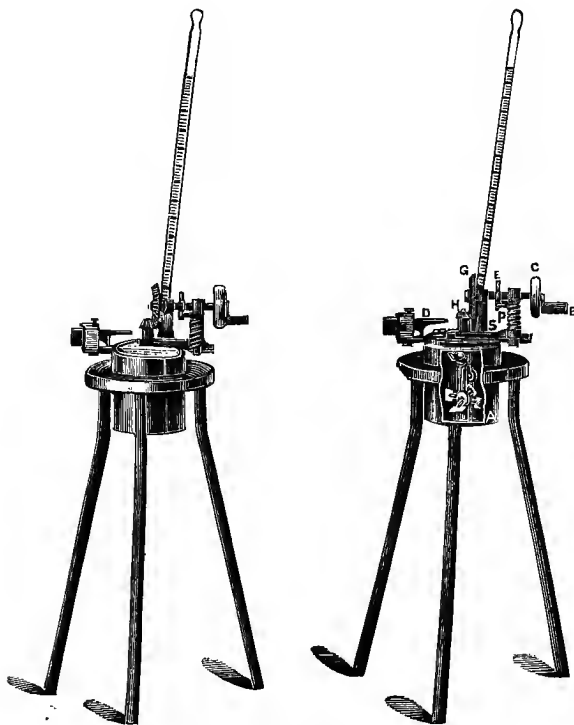


FIG. 58. Gray's Flash Point Apparatus.

observers get very different results, which are due to differences in the construction of the test apparatus, the quantity of air in proportion to the oil used in the apparatus, and one or two minor differences which affect the flash point as determined by the closed test. With the open test these differ-

ences are not produced. It makes but little difference with what apparatus the test is carried out, whether with large or small quantities of oil, providing the rate of heat is not too rapid.

The best instrument for the determining of the flashing point of lubricating oils by a close test is Gray's, shown in Figure 58. This instrument consists of a brass cup of same size as that used in the Government standard petroleum tester, *viz.*, 2 in. in diameter by  $2\frac{2}{10}$  in. in depth. A line cut round the inside of the cup,  $1\frac{1}{2}$  in. from the bottom, indicates the height to which it is to be filled in testing oil. The cup is closed by a tightly fitting cover, through the centre of which a small shaft or spindle passes to the bottom of the cup, carrying two sets of stirrers, one of which is below the surface of the oil, the other in the vapour space above. On the top of the spindle, above the lid, a small bevelled wheel with a milled edge is fixed, which gears with a vertical bevelled wheel on the inner edge of a horizontal shaft carried on two standards fixed on the lid, and terminating at the outer end in a disc of non-conducting material in which is fixed a handle for rotating the shaft. There are four openings in the lid. Through one a thermometer is inserted to indicate the temperature of the oil. The remaining three provide means of producing the flash. One of these is immediately in front of the small tilting lamp which ignites the gases. The other two, one on each side, admit air to produce the explosive mixture. These parts are normally closed by the loose, three-quarter disc, flat cover, provided with openings which, when the cover is turned one quarter round, coincide with the ports in the fixed lid.

In using this instrument, the cup is filled to the mark with the oil to be tested. The test lamp is lighted; the flame being adjusted to about  $\frac{1}{8}$  inch in size. Heat is

applied below the oil cup by a gas or spirit flame, either direct or through the medium of a sand bath, and the temperature of the oil raised at the rate of  $10^{\circ}$  to  $15^{\circ}$  F. per minute; the stirrers being turned at short intervals during the process of heating. When nearing the point at which the oil is expected to flash, it is advisable to reduce the rate of heating in order that observations may be more accurately made.

The test for flashing point is made by drawing the horizontal shaft, which has about  $\frac{1}{8}$  inch end play, outwards, which puts it out of gear with the stirrers and in gear with the sliding cover, which, being partly turned, opens the ports and tilts the flame of the test lamp into the centre one. If gas is present in sufficient quantity, a slight flash with an explosion is produced; if not, the ports are automatically closed, the heating is continued, the stirrers being used, and the test for "flash" repeated at intervals till the flash is observed, the temperature at which this occurs being taken as the flashing point of the oil.

7. *Evaporation test.* This is carried out by weighing into a watch glass a small quantity of the oil, exposing it in an oven to a temperature of  $212^{\circ}$  F. for twenty-four hours (or in the case of the cylinder oils to  $350^{\circ}$  for five hours), then reweighing it. A good sample of oil should not lose more than from 0.25 to 0.5 per cent., and if a sample should lose more than 1 per cent. it ought to be regarded with suspicion.

Archbutt has devised a method of ascertaining the rate of evaporation of cylinder oils, which is said to give reliable and very accurate results. This is carried out in a piece of apparatus specially designed for it. Through a hot air oven he carries a piece of iron tubing  $\frac{7}{8}$  of an inch in diameter; this has fitted in it a glass tube which is just big enough to fit it tightly. In the middle of this glass tube is placed a platinum or porcelain boat, containing a weighed quantity

(0.5 to 1 gramme) of the oil to be tested. In the upper part of the oven is a coil of pipes, through which a current of air at the rate of two litres a minute can be passed. This coil is connected with the glass tube containing the boat of oil, so that the air is heated to the temperature of the oven before it goes over the oil. After an hour's exposure to the current of hot air, the boat of oil is taken out, reweighed, and the loss of weight noted.

Of the two varieties of cylinder oils the filtered oils lose least, the amount of loss averaging less than 0.5 per cent., although samples losing about 0.75 per cent. are met with. The dark natural oils lose most, the loss in some cases being as much as 5 per cent., while occasionally samples of such oils may be met with which actually gain weight.

Particulars of this test are often left out of chemical reports on lubricating oils, but they might be inserted with advantage.

The last three tests are only applicable to mineral or mixed lubricating oils, as the particular properties examined by these tests are invariable as regards each particular fatty oil, while they vary in every different make of mineral or hydrocarbon oil.

As a rule, the application of the tests described above will usually be found to be sufficient for an oil dealer or consumer to obtain a good idea of the quality of the sample to be tested. There are, however, a few other useful tests that may from time to time be found of service in the examination of oils for adulteration or otherwise.

8. *Valenta's acetic acid test.* If equal volumes of glacial acetic acid and a fat oil be mixed together and then heated, the two liquids will amalgamate together and a clear solution will be obtained. On allowing to stand, a cloud will in a short time make its appearance in the liquid. Valenta discovered that this cloud makes its appearance at

different temperatures in different oils, the "turbidity temperature," as it is called, being the point to be ascertained. Valenta's work has been more or less confirmed by subsequent observers, but still there are differences in the recorded observations which serve to show that there are several modifying circumstances in the application and carrying out of the test which must be taken into account. The strength of the acid is important. The temperature at which the turbidity is obtained is distinctly higher with a weak than with a strong acid; a dry test tube or glass is necessary to obtain satisfactory results. Rinsing with water must be avoided, as the small quantity of water left in the glass will affect the subsequent tests. Slight differences are also observable by variations in the manner in which the test is made.

The author has obtained the following results with this test, using 5 cc. of the acid and oil, very convenient quantities, in a wide test tube, the thermometer being immersed in the oil during the whole of the operation:—

Oil.	Turbidity Temperature Centigrade.	Oil.	Turbidity Temperature Centigrade.
Colza . . . . .	99	Rape . . . . .	90
Castor . . . . .	Soluble at Ordinary Temperature	Ground Nut . . . . .	72
Neatsfoot . . . . .	85	Lard . . . . .	76
Cotton Seed . . . . .	63	Olive . . . . .	76
Sperm . . . . .	85	Whale . . . . .	71
Oleic Acid . . . . .	Soluble at Ordinary Temperature	Ox. . . . .	48

For further details, see a paper in the *Journal of the Society of Chemical Industry* for January, 1887.

9. *Hubl's iodine test.* If an oil is mixed with a small quantity of an alcoholic solution of iodine, the latter gradually

becomes decolorised. This has been found to be due to the fatty acid of the oil combining with the iodine and forming compounds with it. The fatty acids present in oils belong to three distinct series: One, the stearic series (better known to chemists as the acetic series); this series does not combine with iodine, so that the larger the proportion of the glycerides of these acids there is in an oil, the smaller quantity of iodine with which they will combine. The second series of fatty acids is the oleic series; each of these is capable of combining with iodine in the proportion of two atoms of iodine to one molecule of fatty acid. The third series is sometimes called the tetrolic or linolic series; these absorb four atoms of iodine for each molecule of fatty acid, and as the acids of these series are characteristic of drying oils, it follows that linseed and other drying oils absorb more iodine than any other class of fatty oils.

Based on these principles is founded Hubl's iodine test, which has for its object the determination of the amount of iodine absorbed by an oil. It is carried out as follows: Five solutions are prepared. 1st, a solution of 25 grammes of iodine in 500 cc. of alcohol; 2nd, a solution of 30 grammes of mercuric chloride in 500 cc. of alcohol; 3rd, a solution of 10 grammes of iodide of potassium in 100 cc. of water; 4th, a solution of 24·8 grammes sodium thiosulphate (hyposulphite) in 1000 cc. of water (each cc. of this is equivalent to 0·0127 gramme of iodine); 5th, a solution of 2 grammes of starch in 100 cc. of water.

Two grammes of the oil are weighed out and dissolved in 100 cc. of chloroform, and from 10 to 20 cc. of this solution are taken, for drying oils 10 cc. are sufficient, for other oils 20 cc. Twenty cc. of the iodine solution and 20 cc. of the mercury solution are added, and the mixture allowed to stand for one and a half to two hours; equal quantities of the chloroform, iodine, and mercury solutions are also



mixed together to make a blank test. At the end of the time, 20 cc. of the iodide solution and 20 cc. of the starch solution are added, and the mixture titrated with the sodium thiosulphate solution, until the blue colour is discharged. The difference in the quantity of the sodium thiosulphate solution used in the two experiments, the blank test and the one with oil, represents the quantity of iodine absorbed by the oil. The quantity of iodine absorbed by 100 parts of oil is called the "iodine equivalent".

10. *Mills' bromine test* depends on the same principle, only a solution of bromine is used instead of the iodine solution, the excess of bromine being measured colorimetrically. The following table gives the iodine and bromine equivalents of a number of the fatty oils:—

Oil.	Iodine Equivalent.	Bromine Equivalent.
Linseed Oil . . . . .	158	76
Cotton Oil . . . . .	106	50
Thickened Cotton Oil . . . . .	50·2	31·4
Rape Oil . . . . .	100	69·4
Thickened Rape Oil . . . . .	45·6	28·5
Castor Oil . . . . .	84·4	58·5
Olive Oil . . . . .	82·8	54
Lard . . . . .	59	37·2
Tallow . . . . .	40	28
Coconut Oil . . . . .	8·4	5·7
Palm Oil . . . . .	51·5	34·7
Hypogæic Acid . . . . .	100	63
Oleic Acid . . . . .	89·8	56·5
Ricinoleic . . . . .	85	53·5
Linoleic Acid . . . . .	201·5	126·9

Hydrocarbon oils are also capable of absorbing iodine and bromine, the quantity absorbed being dependent upon the proportion of olefins the oils contain. Paraffins have no affinity for iodine or bromine: hence American oils, containing as they do more paraffin than Scotch shale oils, absorb less than the latter as a general rule. The following table gives a few iodine and bromine equivalents of petroleum and shale products:—

Name of Oils.	Iodine Equivalent.	Bromine Equivalent.
Scotch Shale Oil, 886 . . . . .	23.5	14.7
"    890 . . . . .	20.6	12.9
"    908 . . . . .	23.2	14.5
"    863 . . . . .	29.2	18.3
"    873 . . . . .	26.0	16.3
American Petroleum Oil, 905 . . . . .	9.7	6.1
"    916 . . . . .	7.8	4.9
"    911 . . . . .	8.1	5.1

11. *Hehner's bromine test.* Mr. O. H. Hehner describes (*Analyst*, 1895, p. 50) the following gravimetric method of determining the bromine absorption of oils: A small wide-mouthed flask is carefully weighed and from 1 to 3 grammes of the fat introduced into it. These are dissolved in 10 cc. of chloroform, and then pure bromine is added, drop by drop, until the bromine is decidedly in excess. Both the chloroform and the bromine must be previously tested in a blank experiment to make sure that they leave no appreciable residue on heating. The flask and its contents are then heated on the water bath till most of the bromine is driven off, a little more chloroform is added and the mixture again heated, the chloroform vapour helping to drive out the excess of bromine. Then the flask and its contents are placed in an air bath regulated for about 125° C., and kept there until repeated weighings show their weight to be constant: this takes several hours. Some acrolein and hydrobromic acids escape during the drying. The increase in weight is the amount of bromine absorbed. This process gives very satisfactory results: the writer has obtained the following figures from different oils:—

Menhaden Fish Oil . . . . .	43.6 per cent.
Pale Seal Oil . . . . .	87.6 "
Pale Whale Oil . . . . .	4.6 "
Scotch Cod Oil . . . . .	59.9 "
Japan Fish Oil . . . . .	123.3 "
Arctic Sperm Oil . . . . .	59.9 "
Straw Seal Oil . . . . .	137.2 "
Olive Oil . . . . .	51.3 "
Lard . . . . .	40.6 "
Butter . . . . .	21.6 "
Castor Oil . . . . .	43.7 "
Linseed Oil (Raw) . . . . .	114.2 "
"    (Boiled) . . . . .	60.0 "
"    " . . . . .	112.0 "

12. *Elaidin test.* For testing the purity of olive oil there is scarcely a better process than the elaidin test or Poutet's test, which was first devised in 1819. The best method of carrying it out is that described by Archbutt (*Jour. Soc. Chem. Ind.*, p. 306). The test solution is made by dissolving 18 grammes of mercury in 15.6 cc. of strong nitric acid of 1.42 specific gravity. The solution is best effected in a glass tube kept cool by immersion in water. The test solution will have a green colour, and as long as it has this colour it is in fit condition to use.

Archbutt used 96 grammes of oil to 8 grammes of the test solution, but 50 cc. of oil to 2 cc. of solution is a very convenient quantity to take. The oil and solution are mixed together and allowed to stand for some hours, being stirred up at intervals if required. The time taken to solidify the oil is noted, the consistence of the solid mass and also its colour.

Archbutt gives the following particulars of results obtained by this test:—

Oil.	Time to Solidify.	Colour of Mass.	Consistence.
Olive . . . .	60 minutes . .	Canary Yellow .	Hard and impene- trable.
Oleic Acid . . .	50 " . . . .	Lemon Yellow .	Penetrable.
Nut . . . . .	60 to 90 minutes	Lemon . . . .	Soft.
Neatsfoot . . .	180 minutes . .	" . . . . .	Penetrable.
Rape . . . . .	More than 6 hours	Deep Orange .	Apparently solid, miscible with water.
Bottlenose . . .	160 minutes . .	Deep Lemon . .	Soft.
Southern Sperm	Not solid, but thick in 6 hours	Orange . . . .	Buttery.
Ground Nut . . .	...	" . . . . .	Soft.
Cotton . . . . .	...	" . . . . .	Turbid, fluid.
Niger . . . . .	...	" . . . . .	Thin fluid.
Cod Liver . . . .	...	Blood Red . .	Fluid.
Castor . . . . .	...	Lemon . . . .	Like Oil.
Sesame . . . . .	...	Orange . . . .	Thick but fluid.
Menhaden . . . .	...	Dark Red . . .	Turbid.

13. *Molecular weight of fatty acids.* Occasionally it is desirable, for the purpose of distinguishing between various oils, to determine the molecular or combining weight of the fatty acids obtained from them. This is done as follows: 10 grammes of the fatty acid are weighed out, dissolved in methylated spirit, a little alcoholic solution of phenol phthalein added, and then titrated with normal caustic soda until a permanent pink coloration is obtained.

To calculate the combining weight, multiply the number of cubic centimetres of caustic soda used by 0.04, which gives the weight of caustic soda needed to combine with the 10 grammes of oil. Then divide this number into the weight of oil taken and multiply the result by 40, when the combining weight will be obtained.

14. *Melting point of fats.* The most convenient way of ascertaining the melting point of fats is that known as the capillary tube method. This is carried out in the following manner: A piece of thin glass tubing is drawn out at one end into a capillary tube. The fat or substance whose melting point is required is heated in a small glass beaker until it is just melted, and the end of the capillary tube dipped into the melted fat; some will be taken up. The tube is withdrawn and placed on one side for some time to cool, to allow the fat to solidify. The capillary tube is next tied to the stem of a thermometer in such a way that the bulb and capillary tube where the fat is are close together. A beaker of cold water is placed on a sand bath and heated by a bunsen burner. The thermometer is suspended in the centre of the water. As long as the temperature remains below the melting point of the fat, the latter will be opaque and stationary, but when the melting point is reached then the fat becomes transparent and will rise in the tube. When this event is noticed, the temperature is read off on the thermometer, and this gives the melting point of the fat.

15. *Reichert's test.* Some fats, notably coconut oil, palm nut oil, butter, whale oil, contain fatty acids which are soluble in water, and, when distilled along with water, volatilise and distil over, the distillate having an acid reaction. It has been found possible to employ this property as a test in the examination of oils, and from having been originally devised by Reichert, is known by his name. Since its first introduction other analysts have published sundry modifications in the manner of carrying it out.

Reichert's test is carried out in the following manner : 2·5 grammes of the oil or fat are thoroughly saponified by boiling with 25 cc. of a normal or nearly so alcoholic solution of caustic potash, in the same manner as is adopted for the Koettstorfer. After the saponification is complete, boil until all the alcohol has been boiled off, then dilute with water, add sufficient dilute sulphuric acid to decompose the soap, add water, if necessary, to bring up the volume to 75 cc. Then distil in a retort until 50 cc. have come over, taking care to avoid bumping over of the liquid. Should the distillate be cloudy or show the presence of fat globules filter it, washing the filter with warm water. Then titrate the clear distillate with decinormal caustic soda, using phenol phtalein as an indicator, noting the number of cc.'s of the alkaline solution required.

The following are a few figures obtained with this test :—

Fat or Oil.	No. of cc.'s of $\frac{N}{10}$ KOH used.
Butter . . . . .	12·5 to 15·2
Coconut Oil . . . . .	3·5
Palm Nut Oil . . . . .	2·5
Palm Oil . . . . .	0·8
Whale Oil . . . . .	3·7
Sperm Oil . . . . .	1·7
Cotton Seed Oil . . . . .	0·3
Castor Oil . . . . .	1·4
Thickened Rape Oil . . . . .	1·2
„ Cotton Seed Oil . . . . .	1·3

## OIL TESTING MACHINES.

For the purpose of obtaining a greater knowledge of the practical working of oils in the lubrication of machinery than is possible by means of the tests given above, mechanical tests have been devised from time to time, and quite a number of these oil testing machines are now in use in various oil works and machinery establishments in the country, many having been specially designed for the users' own purposes, and have not been put in the market.

Practically, only three are now in much use; these are the Ingram & Stapfer, the Thurston and the Thomas.

In order to determine precisely what oils are adapted to any special purpose, or to ascertain what uses an oil is best fitted for, it is necessary to make an examination of the lubricant while it is working under the specified conditions. That is to say, the oil should be put upon a journal of the character of that on which it is proposed to use it, and subjecting it to the pressure proposed, and running it at the speed at which the journal is expected to attain, its behaviour under these conditions will then show conclusively its adaptability to such a purpose.

While running it is necessary to measure the friction produced and to determine its efficiency, as well as to be able to note its durability and the rise in temperature of the bearing. These qualities being determined and recorded, all is known of the oil that is needed to determine its lubricating power, and its value for the purpose intended.

The Ingram & Stapfer Machine is made by Messrs. W. H. Bailey & Co. of Salford. This machine is represented in Figure 59, and is made in two sizes: a small size for ordinary oil testing, and a large size adapted for the testing of heavy machinery oils, railway work, etc., intended for use in large bearings, carriage and waggon axles.

This machine consists of a bed-plate having upon it a

pair of standards carrying a short length of shafting, upon which is a testing journal fitted with two brass steps, the speed at which the shaft is driven being about 2000 revolutions per minute, but can be regulated. The friction is brought to bear by levers, and weights somewhat after the manner of a friction brake, as shown in the drawing, acting on the steps. On the top step is a thermometer for indicating any increase in temperature caused by the friction. A dial indicates the number of revolutions that the shaft makes during the test. The machines used for testing light oils

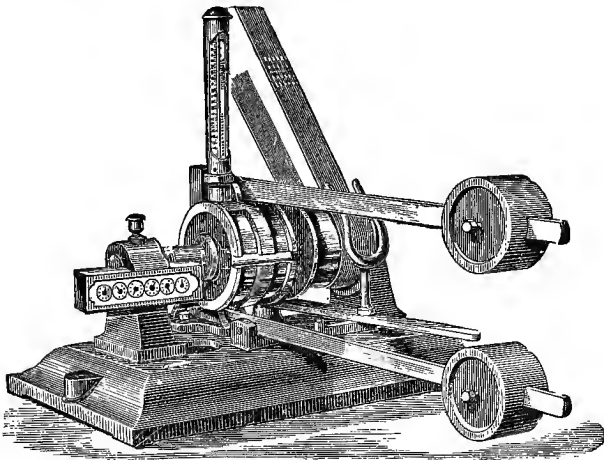


FIG. 59. Ingram & Stapfer Oil Testing Machine.

have the friction journals, where the oil is tested, three inches in diameter; those for testing tallow and heavier oils are of larger diameter (six inches).

This machine is used in the following manner: The top and bottom steps should be well cleaned before each experiment, and kept free from dust; the bearings of the spindle should be well oiled to prevent friction in the wrong place. It should run half a day well oiled before a test takes place when first fixed.

The oil must be measured in a small glass tube (an homœopathic bottle will do, or a hole in a small key, if it is smooth inside). In emptying its contents, the measure should be warmed over a flame to enable the oil to flow freely from it. The measure should be well cleaned out each time. Two drops only have often been used, and a glass pencil dipped into the oil, and then held until the drops accumulate, will be found useful, but a definite weight or measure is the best.

The thermometer should, as far as possible, always indicate the same temperature at starting. It is found that 150° F. is the best to try all oils to, if their lubricating power is to be measured, and the machine should always be driven until that temperature is indicated, and then immediately stopped. The machine should be put in a place not liable to sudden changes of temperature.

When a temperature of 150° F. has been reached (the speed index showing zero at the start), it will then be seen what number of revolutions is taken to produce the temperature (see table below). The speed should be about 1500. A uniform speed should be maintained in all tests.

After obtaining figures in this way from an oil, stop the machine and let the oil remain on the machine, and in twelve hours after see how soon 150° F. can be obtained. The second experiment will indicate which of the two oils is the inferior on machinery, when stopped, by reason of its tendency to gum through oxidation.

Should the oil indicate or have any tendency to gum or become thick by exposure to the air, this will be indicated by a smaller number of revolutions being required to produce the temperature of experiment.

Another method of running this machine is to run it for a definite number of revolutions and for a fixed time, say one hour and a half, and notice the temperature which has



been reached in that time. The following are some figures which have been obtained with this machine :—

Name of Material.	Heat Developed in 1½ Hours.
Castor Oil . . . .	158
Resin Oil . . . .	155
Tallow or Animal Oil . . . .	141
Rape Oil . . . .	148
Lard Oil . . . .	146
Olive Oil . . . .	143
Sperm Oil . . . .	133
Mineral Oil . . . .	121
” ” . . . .	117
	Heat Developed in 1 Hour.

Olive oil when run for 20,000 revolutions gave a temperature of 175° F. A mixed lubricating oil gave for the same number of revolutions 165° F.

A sample of ox oil required the machine to be run for 13,000 revolutions to reach the temperature of 200° F. when fresh, and only 11,700 revolutions when the oil has been on the bearing for twelve hours. Similarly a sample of sperm oil ran for 16,900 revolutions when fresh and 13,000 after exposure.

The relative wearing qualities of the animal and vegetable and mineral oils are observable on this machine. The former can be run for one and a half to two hours, while the latter are exhausted after half an hour's run.

The Thurston Oil Testing Machine, shown in Figures 60 and 61, is a very ingenious one. It is the invention of Professor R. H. Thurston, of the Technical College at Hoboken, New Jersey, United States. The Thurston oil tester consists of a small shaft running in two bearings, carrying a journal on one end. This journal is grasped by two brass steps which are in connection with a pendulous weight. These brass steps are forced against the journal by means of a screw which compresses a coil spring. The amount of this pressure is indicated on a scale like that of a spring balance. A “bob” at the end of the pendulous arm gives the weight necessary to resist deflection.

The angle of deflection is measured on an arc or quadrant graduated in such units that the figures which may be read off give, not only the angle of deflection, but also the coefficient of friction. A thermometer on the top brass gives the temperature in a manner similar to that of the Stapp test. This machine is used much in the same way as the tester previously described. It is used in the engineering

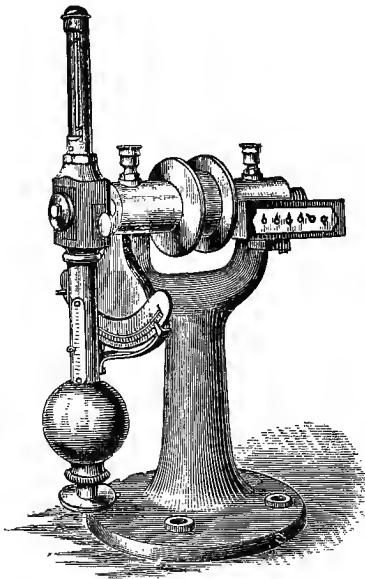


FIG. 60. Thurston's Oil Testing Machine.

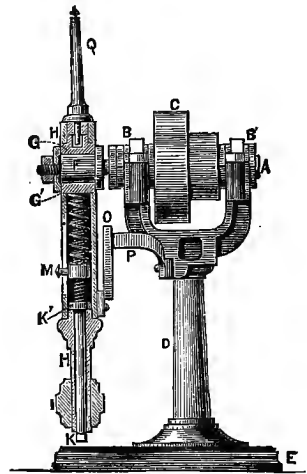


FIG. 61. Sectional View of Thurston's Oil Testing Machine.

shops of the United States Navy and many railway works. The small machine (Figure 61) is for testing the ordinary range of oils, while a large machine is made for heavy oils for heavy bearings.

This machine is adapted for a wide range of pressures, as is seen in the index plate in front of the pendulum, where the large figures represent the total pressures on the journal,

and those opposite the corresponding pressures per square inch.

To obtain the best results it is essential that each test should be made at the pressure under which it is proposed that the journal on which it is to be used shall be run.

The periphery speed of the journal should be equal to the maximum periphery speed of the shafting or journals on which the selected oil is intended to be used.

Figure 61 is a sectional view with index lettering.

The figures on the arc P, traversed by the pointer attached to the pendulum, are such that the quotient of the reading of the arc, divided by the total pressure read from the front of the pendulum at M, gives the "coefficient of friction," *i.e.*, the proportion of that pressure which measures the resistance due to friction.

To determine the lubricating qualities of an oil remove the pendulum HH (see sectional illustration, Figure 61) from the testing journal GG, adjust the machine to run at the desired pressure by turning the screw head K, projecting from the lower end of the pendulum, until the index M in front of the pendulum shows the right pressure; adjust it to run at the required speed. Throw out the bearings by means of the two thumb screws on the head of the pendulum in the small machine, or by setting down the brass nut immediately under the head in the case of the large machine. Carefully slide the pendulum off the testing journal GG, and see that no scratching of journal or brasses takes place. Place a few drops of the lubricant to be tested on the journal, replace the pendulum, and set the machine in motion, running it a moment until the oil is well distributed over the journal. Next stop the machine, loosen the nut or the cams which confine the spring, and, when it is fairly in contact and bearing on the lower brass with full pressure, turn the brass nut or the cams fairly out of contact, so that

the spring may not be jammed by their shaking while working. Now, start the machine again, and run until the behaviour of the oil is determined. At intervals of one or more minutes, as may prove most satisfactory, observe and record the temperature given by the thermometer Q, and the reading indicated on the arc P of the machine by the pointer O. When both readings have ceased to vary, experiments may be terminated.

Remove the pendulum, first relieving the pressure of the spring, and clean the journal and brasses with great care from every sign of grease, and be especially careful not to leave a particle of lint on the surfaces.

A comparison of the results thus obtained with several oils will show their relative values as reducers of friction.

Note should be made of the following points:—

Temperature before and at end of experiment, showing increase.

Pressure.

Number of revolutions.

Duration in time of test.

Readings on the arc of the machine.

To determine the liability of the oil to gum, allow the machine to stand with the journal wet with oil for twelve or twenty-four hours or more, as may be found necessary. Then start up and run a few moments until the reading on the arc P, having fallen to a minimum, begins to rise again, then stop at once. Compare the minimum coefficients thus obtained from the several oils to be examined. That which gives the smallest figure will be least liable to gum during the period of time given to the test.

To determine durability, proceed as in determining the lubricating quality, and apply, say, a drop for each two inches length of journal, then start the machine. When the friction, as shown by the pointer O, has passed a minimum

and begins to rise, the machine should be carefully watched, and should be stopped either at the instant the friction has reached double the minimum, or when the thermometer indicates 212° F. This operation should be repeated until the duration of each trial becomes nearly the same. An average may then be taken either of the time, of the number of revolutions, or of the distance rubbed over by the bearing, which average will measure the durability of that lubricant.

Next carefully clean the testing journal and proceed as before with the next oil to be tested.

A lubricant is valuable in proportion to its durability and its freedom from tendency to gum, and in proportion as it exhibits a low measure of friction. In making comparisons, always test the standard as well as the competing oils on the same journal, and under precisely the same conditions. An approximate value by which to compare the oils can be calculated, based on the assumption that they will have a money value proportionate to their durability and to the inverse ratio of the value of the coefficient of friction. Thus, suppose two oils to run, the one ten minutes and the other five minutes, under a pressure of one hundred pounds per square inch, and both at the same speed, and suppose them to give, on test for friction, the coefficients 0·10 and 0·6 respectively, their relative values might be taken at  $\frac{10}{6}=1$  and  $\frac{5}{8}=0\cdot625$ . If the first is worth, say, 100 pence, the second should be worth 62 pence. In many cases, however, the same quantity would be applied by the oiler, whatever oil might be used, and their values to the consumer would be the inverse proportion of the values of their coefficients of friction, *i.e.*, as six in the above case is to ten, thus showing that it would be cheaper to use the latter if the cost is anything less than its relative value.

The following tables show some tests of oil made with this machine :—

Oil.	Pressure in sq. ins.	Time of Run.	Increase in Temperature.	Readings on Arc.	Revolutions.
		Mins.			
Sperm . . . . .	75	85	35	3 to 6.5	27,870
Lard . . . . .	75	76	55	5 to 10.0	24,500
Lubricating Oil .	75	93	95	8 to 16.0	25,720

COEFFICIENTS OF FRICTION AND ENDURANCE OF LUBRICANTS.

Name.	Pressure.	Endurance.	Rise in Temperature.	Co-efficient.
	Lbs.	Min.	F.	
Sperm (Winter) Oil . . . . .	8	111	230	0.13
	16	29	225	0.10
	48	9	195	0.08
,, (Summer) Oil . . . . .	8	165	170	0.13
	16	33	215	0.11
	48	7	265	0.10
Lard Oil . . . . .	8	77	175	0.16
	16	27	250	0.12
	48	11	260	0.07
Neatsfoot Oil . . . . .	8	106	205	0.15
	16	31	275	0.10
	48	6	190	0.10
Olive Oil . . . . .	8	83	170	0.13
	16	41	245	0.10
	48	14	240	0.06
Cotton Seed Oil . . . . .	8	107	185	0.16
	16	45	275	0.12
	48	12	310	0.07
Palm Oil . . . . .	8	49	195	0.17
	16	15	235	0.13
	48	9	295	0.07
Castor Oil . . . . .	8	45	160	0.19
	16	35	180	0.11
	48	11	375	0.07
Fish Oil (Cod) . . . . .	8	40	200	0.15
	16	14	175	0.12
	48	9	220	0.07
Crude Mineral Oil . . . . .	8	129	105	0.10
	16	97	285	0.10
	48	5	270	0.10

The bearings were run dry.

The speed of the testing journal was 750 feet per minute.

The coefficient of friction is obtained by dividing the readings on the arc by the total pressure.

A comparison of the results thus obtained with

several oils will show their relative values as reducers of friction.

Steam cylinder lubricants are tested on bearings heated to a temperature corresponding to any desired steam pressure. When the maximum temperature has been attained, the flame is removed, and the behaviour of the oil noted as the temperature falls to  $212^{\circ}$  F., which corresponds to atmospheric pressure or to zero on the steam gauge. Any effervescence or excessive friction at the higher temperatures condemns the lubricant. This machine is made by Messrs. W. H. Bailey & Co.

The Thomas "Friction" Oil Testing Machine is shown in Figure 62. The testing portion of this machine, seen in front of the drawing, consists of an annular disc containing a recessed groove, which is made exactly one square centimetre in size and one metre in circumference, so that the circumferential speed can be easily ascertained. This annular disc is driven by the arrangement also shown in the drawing. A belt for the driving shaft is attached to a pair of fast and loose pulleys on a counter shaft. On this is placed a set of three cone pulleys, and these are in gear with a similar set on the shaft of the testing disc. By this arrangement the speed can be varied to almost any desired extent, so that oils that are to be used on quick-running machinery can be tested under similar conditions as to speed, while heavy oils for heavy slow machinery can be tested at the slow speeds at which they are to be used. This is an advantage not possessed to the same extent by other testing machines.

In the annular recess in the disc works a friction block. This is pressed down by the weight shown hanging from the bracket. This weight can be varied to any extent, from, say, 10 lbs. to 100 lbs., and whatever weight is used is the pressure per square centimetre.

Now the revolution of the disc tends to carry the friction

block out of the centre line until it reaches a point where the two forces, the friction and the weight, tend to neutralise

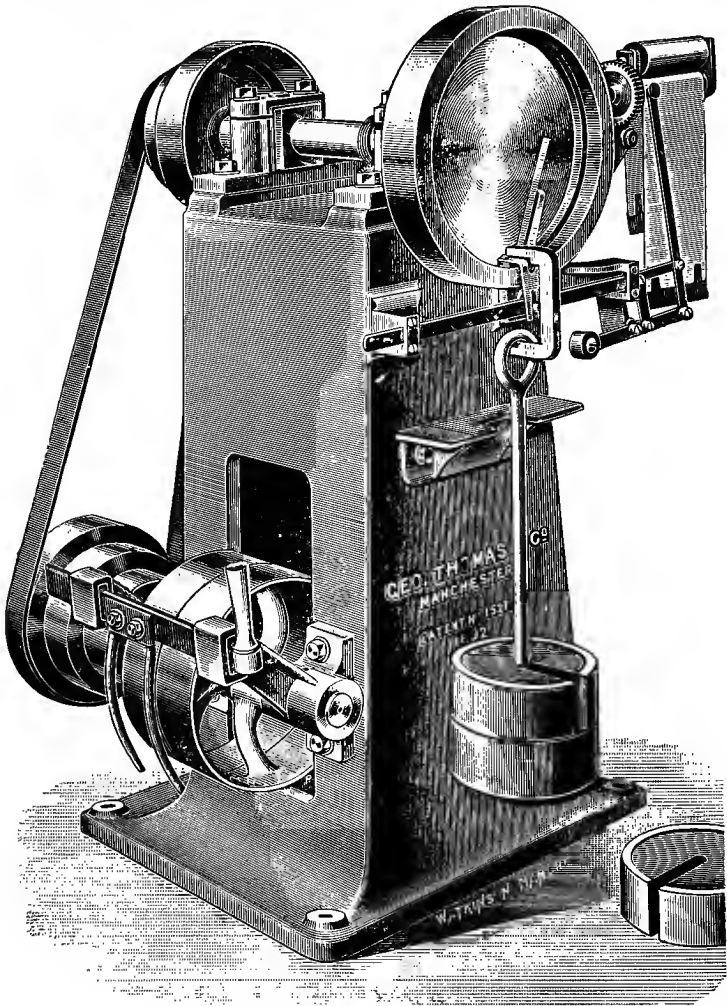


FIG. 62. Friction Oil Testing Machine.

each other, and there it will remain more or less stationary. This point will depend upon the speed of revolution, the



weight or pressure exerted on the friction block, and upon the quality of the lubricant. In the latter case, the better the oil or lubricant used, or the better the degree of lubrication, the less is the block drawn out of the centre line. In the machine this factor, which represents what is known as the coefficient of friction, is made to manifest itself in a visible and recorded form, by an arrangement of levers which carry a pencil point, and draw a line on a paper ribbon, which is drawn between a pair of rolls, seen on the right of the drawing, at a low rate. If the lubrication is good, a fairly straight line is drawn, while if it varies much, then a zigzag line is drawn; while the amount of the coefficient is shown by the position of the line in reference to the datum line on the paper ribbon. It is in thus recording the amount of the coefficient of friction that this machine differs from all hitherto made. Some diagrams made by this machine are given in Figure 63, and they show very clearly the relative value of different oils for the same purpose.

A thermometer, which has its bulb inserted in a hole in the friction block, shows any and what increase of temperature takes place. This a feature of some importance.

By means of a Bunsen burner the annular disc may be heated up to any required degree of temperature. This is a feature of this machine, which will be found extremely useful in testing cylinder oils, and such can be tested at temperatures ranging to 350° F., a temperature which is sometimes attained in the cylinders of high pressure engines.

There is one feature in this machine whereby it has advantages over some others which have been made. The whole of the oil is applied to, and is certain to come into contact with, the friction block. This is not a certainty with other forms of oil testing machines. Then again the effect of varying methods of lubrication may be efficiently tested with this machine. It may be fed with a stated

quantity of oil, and the length of time this will wear may be ascertained, or the feed may be continuous, and the machine run for a stated time to ascertain, under continuous lubrica-

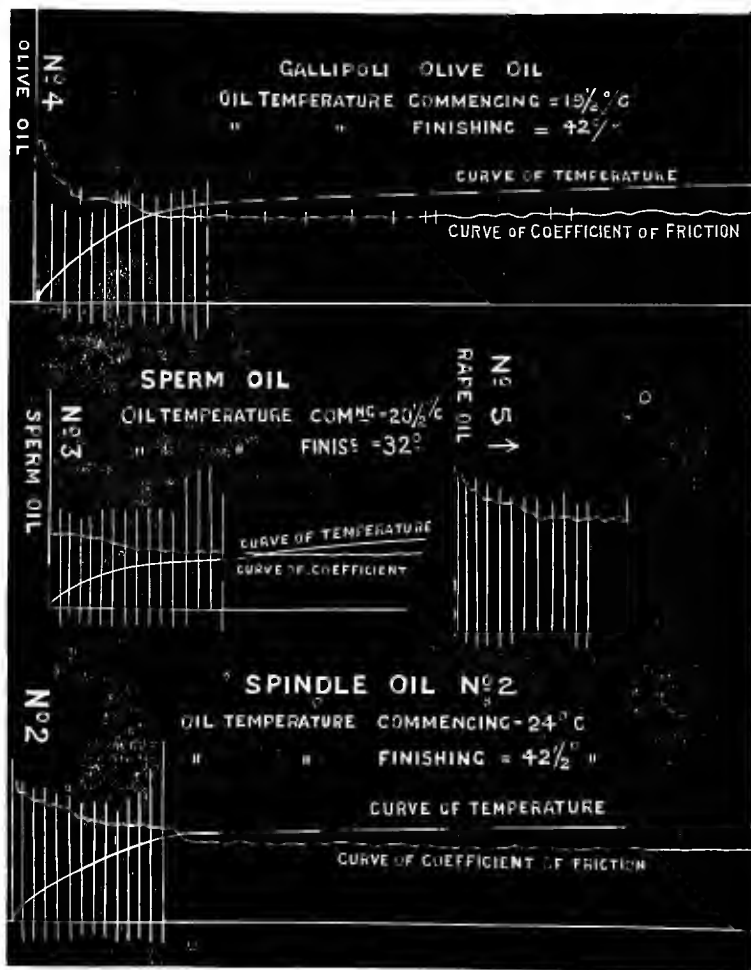


FIG. 63. Diagrams of Oil Tests.

tion, what the coefficient of friction is, and its variation, and what temperature is attained during the run.

A brief notice may be given here of some other forms of testing machines.

*McNaught's*. This consists essentially of a small revolving disc supported on suitable bearings. On this disc a loose one rests, the two faces in contact being carefully and accurately made. By friction the loose disc is carried round by the revolving disc. On the loose disc, however, is fitted a pin which comes in contact with the arm of a lever connected with a graduated balance arm. On this balance arm is arranged a sliding weight, and by moving the weight along the arm a point is reached where the friction between the two discs is counterbalanced by the weight and the loose disc remains stationary. By introducing a few drops of oil between the two surfaces the friction is reduced, and this reduction is measured by a less weight being required to bring the loose disc to rest. With different oils the weight will have to be on different portions of the arm, thus measuring the friction-reducing powers of the oils. This machine does not, however, give entirely satisfactory results.

*Napier's*. Consists essentially of a revolving wheel against the edge of which a brake block is pressed. The amount of friction between the two tends to carry the brake block round; the force which is exerted to effect this can be measured by means of a dynamometer, while the pressure exerted by the brake block can also be measured. By introducing oils on to the revolving wheel the friction is reduced in proportion to the quality of the oil, the reduction of friction being measured by the dynamometer attached to the machine.

*Shaw's* machine is a simple and yet effective machine. This is constructed as follows: A surfaced disc of metal about 4 inches in diameter is mounted on a vertical shaft or spindle which is made to revolve at about 700 revolutions

per minute. The oil to be tested is put on this disc, and a block of metal with a surfaced face is placed upon it, and a cord or chain connected with a spring balance is attached to a pin screwed into the side of the block. A hole is bored through the centre of this block, in which a thermometer is placed, so that all tests may commence at a fixed temperature. When the machine is set in motion, the body or thickness of the oil is indicated on the spring balance, and as the oil thins or gets exhausted by the frictional heat, it rolls off the disc and the balance runs down. If the oil contains nothing but lubricating matter, it will go down to about one pound, which is the friction of the surfaces without oil, but if there is any glutinous ingredient in the oil, the balance will not run down to one pound, but the block will begin to jump or jerk at various indications, from 5 pounds to 1 pound, according to the amount of viscous matter contained in the oil. The presence of gum is also shown by allowing the waste oil to remain on the edge of the disc until cold, when it will be sticky if there is gum, but limpid and soft if pure oil.

Another form of oil testing machine consists of a heavy vertical disc, revolving on a horizontal axle with rather long bearings, very accurately fitted. The wheel is caused to revolve by a known force, and the lubricating power of a sample of oil is measured by the number of revolutions which the wheel will make.

## CHAPTER VII.

### LUBRICATING GREASES.

THESE greases are made of a very great variety of materials, good, bad, and indifferent. For some kinds anything is thought to be good enough, while there are others made of good materials for lubricating certain special bearings where no other kind of lubricant can possibly be used.

To these greases fancy names are often given, more or less descriptive of the particular purpose for which the grease is to be employed, while some of the names are altogether fancy, and are not indicative either of the use or the composition of the grease.

Most lubricating greases are made by treating a grease oil or fat with an alkaline body, when a soap is made, which amalgamating with the rest of the oil makes the latter stiff and greasy. The two alkalies chiefly used are lime and soda, the former when crude, rough greases are required, the latter when better qualities are desired, although some greases do not contain any alkali at all. Sometimes what are known as fillings are put in. These consist of such bodies as powdered gypsum, mica, French chalk, black lead, etc., some of which add to the lubricating value of the grease, while others do not and are put in to make the grease apparently stiffer.

The following fats and oils are used in making these lubricants:—

## PALM OIL.

This material is used in making the best qualities of loco greases. For this purpose the poorer qualities of palm oil, which are rich in free acid, give rather better results than the better qualities, because, during the process of making, the acid enters into combination with the alkali used and forms a soap which amalgamates with the remainder of the oil and any other oil substance added to form the grease. If the palm oil contains little free acid it is obvious that this saponifying action cannot take place, and the formation of grease takes place but imperfectly.

Tallow is frequently put into greases ; of course only the commoner qualities are used, as the finer qualities have greater value as soap stock.

OIL FOOTS of all kinds are commonly used. They are scarcely usable for any other purpose owing to the colouring matter and other impurities that they contain.

ROSIN OIL is one of the commonest grease materials, and generally the crude grades are thus employed.

## ROSIN OIL.

Rosin is the solid residue which is left behind when the crude turpentine resin from various species of pine trees has been distilled by means of fire heat and steam to obtain the turpentine spirit used by painters. Rosin has a strong acid nature, but its constitution has not yet been fully determined ; some authorities consider that it is a mixture of two isomeric acids known as pinic and sylvic, while other authorities consider that it is the anhydride of abietic acid. It makes its appearance in the form of large homogeneous masses, more or less transparent, varying in colour from pale amber to almost black.

When distilled in a still with fire heat alone or with

the combined aid of fire heat and superheated steam, rosin undergoes decomposition and there are obtained:—

- (1) A watery acid liquor.
- (2) Rosin spirit.
- (3) Rosin oil.
- (4) Pitch or coke.

The operation is conducted in a large iron still connected with suitable boilers of about 2000 gallons capacity, the operation taking about 35 to 36 hours to perform when fire heat alone is used. There are usually obtained 3.1 per cent. of spirit, 85.1 per cent. of oil, 3.9 per cent. of coke, 2.5 per cent. of acid water, 5.4 per cent. of loss.

When superheated steam is used 15 per cent. of rosin spirit, 62½ per cent. of oil, and about 10 per cent. of pitch are obtained. The acid water contains about 12½ per cent. of acetic acid. It is, however, rarely used for making that product. The rosin spirit is refined by treatment with sulphuric acid and soda and redistillation, when it is used as a solvent in varnish making.

The crude rosin oil is a thick, viscid, dark brown coloured liquid which, on standing, deposits crystalline particles. It is certainly acid in nature, has a peculiar and characteristic odour, and a strong bluish bloom. Three varieties of crude rosin oil are collected at different periods of the distillation. These are known as “hard,” “medium,” and “soft”. The character of the oil is also modified by the method of distilling. If the distillation is conducted rapidly, then the “hard” rosin oil is the result. If it is conducted slowly, then “soft” rosin oil is the product. Hard rosin oil is much thicker in consistence than soft rosin oil. It is used to mix with lime for making lubricating greases, while, on the other hand, the soft fluid oil is employed for making greases with lime which are more fluid than those got with hard oil. While rosin oil is mostly sold in the crude condition as it comes

from the still, especially for use in making greases, it is often subjected to a process of refining which consists in treatment with (1) sulphuric acid, and (2) caustic soda and redistillation. In some cases the latter may be omitted. By this treatment rosin oil becomes much paler in colour and freer from any solid particles. By repeated operations, rosin oil of a pale yellow lemon tint and neutral in its character can be obtained. Rosin oil has a very high specific gravity, varying considerably from 0.985 to 1.02. It is thick and viscid, but becomes fluid on heating. When heated it gives off the peculiar odour of burning rosin. Its flash point is very low, usually about 322° F., and the flashing is of a peculiar character, being somewhat scintillating. The fire test also is low, about 400° F. Another peculiar property of rosin oil is its action on polarised light, rotating the ray 30° to the right. In this respect it differs from all other oils. The composition of crude rosin oil is exceedingly complex. It contains members of the olefin, naphthene, benzene, and terpene hydrocarbons. There are also present alcohols, aldehydes, and ketate compounds, as well as acid bodies. The more highly refined rosin oils do not contain the ketate compounds, but comparatively more of the hydrocarbons.

Some of the crude rosin oils exhibit the tendency to dry and become hard when exposed to the air, and hence have been used for making paint. Their use for this purpose is not satisfactory, for the oil does not dry properly, and exhibits the tendency to soften after a long exposure to the air.

The crude rosin oils are largely used for making greases, as will be described later on, while the more refined oils are used for general lubricating purposes, for which purpose they cannot be considered as ideal lubricants unless great care has been taken in refining. They are of an acid character, and have a corrosive action on the bearings of machinery; While being very viscid, they can only be used for lubricat-



ing very heavy shafting and machinery. It should be stated, however, that the best grades of rosin oil are quite neutral, and have no corrosive action on metals. Rosin oil is sometimes added to lubricating oils, but its presence is detected by its high specific gravity and low flash point. It also has a very peculiar and characteristic taste which cannot be mistaken.

Some information about rosin greases will be found below.

The following are some details concerning the various grades of crude rosin oils :—

	“Hard.”	“Medium.”	“Soft.”
Specific Gravity at 60° F. . . . .	0·987	0·992	0·986
Viscosity at 70° F. . . . .	215	305	140
”    100° F. . . . .	65	98	29
”    150° F. . . . .	25	30	16
Flash Point . . . . .	200° F.	220° F.	220° F.
Fire Point . . . . .	230° F.	280° F.	300° F.
Free Acid . . . . .	33·67°/o	19·74°/o	2·82°/o
Hydrocarbon Oil . . . . .	51·90°/o	68·50°/o	90·00°/o
Ethereal Oil . . . . .	14·43°/o	11·76°/o	7·18°/o

#### YORKSHIRE GREASE. BROWN GREASE.

In the woollen industry a large quantity of soap and oil is used in cleansing, milling, and oiling the fibre, yarn, and cloth, during the different processes involved in spinning and weaving wool. Formerly all these products, or rather the waste from them, went into the rivers in the form of soapsuds, and thus helped to pollute them.

Now almost all of the soapsuds of the woollen mills of Yorkshire and other districts are treated with acid and the fat which is liberated used again for various purposes.

The soapsuds are collected in a large tank, calculated to hold a day's supply, say from 6000 to 8000 gallons

of liquor. Usually two tanks, constructed of brickwork or concrete, are provided, one being used to collect the suds, while the suds collected in the other tank are being treated in the manner following: Sulphuric acid, or preferably hydrochloric acid, is added to the suds, and after thoroughly agitating the mixture it is allowed to settle, the fatty matter comes to the top, is skimmed off, and thrown on to large cloth filters to drain. The fatty matter thus collected is known as "magma" or "sake," and the operation of treating the suds with acid is commonly known as "saking," probably a local corruption for "seeking".

This magma is then sent to the grease mills, while the residual liquid is run into the river. This magma is now put into bags, which are piled up in a tank and then weighted, and so left for a night, when a large quantity of water is pressed out. Then the cakes of magma are placed in a hydraulic filter press kept hot by means of steam, and subjected to pressure, when a dark brown greasy product is pressed out, and a more solid matter is left behind in the bags. The latter is known as "sud cake" and is used as manure. The greasy matter referred to is known as "Yorkshire grease".

This body is sometimes used for making soap for wool scouring, but is more frequently distilled for preparing wool oil and stearin.

Yorkshire grease varies a little in appearance, but usually is of various shades of brown, sometimes almost black. It is very sticky, and easily melts, its melting point being  $44^{\circ}$  C. On treatment with soda or potash, it undergoes partial saponification; the soap so produced is not readily soluble in water, and is of very short grain. Its specific gravity at  $15.5^{\circ}$  C. varies from 0.939 to 0.957. So far as my experiments go the lower specific gravity is always associated with a low percentage of unsaponifiable matter.

It contains free fatty acids, unsaponifiable matter, and neutral oils, etc.

The free fatty acids vary with different samples according to the character of soap or fat originally used. The unsaponifiable matter contains cholesterin, derived from the natural fat of the wool, and probably also some of the mineral oil which has been used in the wool batching. The grease also contains water and mineral matter.

The following table shows the composition of four samples of Yorkshire grease examined by the author :—

	1.	2.	3.	4.
Specific Gravity at 15° C. . . . .	0·9391	0·9417	...	0·9570
"          98° C. . . . .	0·8900	0·8952	...	0·8720
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Water . . . . .	0·98	1·21	1·21	0·94
Fatty Acid . . . . .	18·61	24·25	24·25	26·43
Neutral Oil . . . . .	68·62	53·25	30·02	16·86
Unsaponifiable Oil . . . . .	11·68	15·83	44·34	55·77
Ash . . . . .	0·11	0·14	0·18	Trace.
	100·00	99·68	100·00	100·00

The grease is also used in the manufacture of lubricating greases, and it may be of interest in this connection to give its flashing point, etc. :—

Vaporising Temperature . . . . .	° C.	° F.
Flashing Point . . . . .	182	359
Fire Test . . . . .	220	428
	248	478

It makes very stiff greases of high melting point.

ANTHRACENE OIL is a bye-product obtained from coal tar. It is a dark greenish-brown liquid and oily in appearance, the colour varying considerably. It is rather heavier than water, the specific gravity being 1·065 to 1·100. It smells strongly of tar oils; mixed with lime it forms a thick but oily grease. It is a cheap material, probably

the cheapest of all grease stocks, and is therefore much used in making the cheaper qualities of greases.

DARK PETROLEUM OILS are often mixed with greases to give them greater lubricating powers. These oils are of brownish colour, varying in consistency from liquid oils (summer dark) to thick tarry oils (cylinder oils). They are perfectly neutral, and have no power of combining with alkalies. They will dissolve soap when the latter is presented to them in a dry condition. These oils are fairly cheap. In some of the better qualities of greases the filtered petroleum oils, which are of a brown or yellow colour, are employed.

Regarding the use of caustic soda and lime little need be said. The former gives smoother greases than the latter, and its soaps are more easily soluble in the other oils. Lime is the cheaper of the two, and is hence used in making the cheapest greases. It gives stiffer greases than soda, which has a higher melting point.

Of the filling materials used, gypsum, or mineral white, has no lubricating power at all. French chalk possesses some slight lubricating properties, being smooth and soft to the touch. Black lead, or plumbago, is a well-known lubricant, especially for wood.

#### MANUFACTURING RECIPES.

The following details showing the method of making various kinds of lubricating greases will be found of service:—

*Wheel Grease.* Take 5 pounds of quicklime and slake with 20 pounds of water, then sieve well, and stir into the lime paste 4 gallons of "hard" crude rosin oil, then allow it to stand for twelve hours, pour off the water, and stir in 5 gallons of anthracene grease oil. Now heat the mass to 240° F., stir well the whole time, until a good mixture is obtained, then allow to cool to set.

*Tram Grease.* Take 10 gallons of anthracene oil, and

stir in a paste made from 5 lbs. of quicklime, well slaked, and mixed with 5 lbs. ground gypsum, then heat up as before. In heating greases which contain water care must be taken, as they froth a great deal, and hence capacious vessels must be used. Too prolonged heating is to be avoided, as with some greases so doing reduces the stiffness very considerably.

*Hot-neck Grease* Take 20 lbs. of soap, cut in thin flakes, and dry it. Then take 30 lbs. filtered cylinder oil, and 30 lbs. 915 petroleum oil. Mix the two together, and heat to 240° F. Then add the soap and stir well, maintaining the heat until the soap and oil have amalgamated, when the mixture may be allowed to cool down. When cold it will be found to be stiff.

*Axle Grease for Wood.* Take 2 gallons of "medium" rosin oil, and stir in 5 lbs. of quicklime, slaked with 2 gallons of water. Then stand for 12 hours, or until the next day. Pour off any water that may separate. Then stir in 5 gallons of coal tar grease oil and 5 lbs. of powdered black lead. Generally it will be found sufficient to mix the materials cold, but a little heating will make a more homogeneous grease.

*Loco Grease.* A common kind of loco grease can be made from 60 lbs. Yorkshire grease mixed with 20 lbs. summer dark oil, and heated with 6 lbs. quicklime, slaked with 2 gallons of water. The best loco grease is made from palm oil, tallow, seal oil, and soda crystals. The soda crystals are dissolved in about an equal weight of water, and then stirred into a melted mixture of the fats. The proportions used are varied according to the different seasons of the year. In summer a stiffer grease can be used than in winter. This variation is attained by using more palm oil and soda crystals in summer than in winter, while it is the custom to add a little sperm or seal oil in winter. The

proportion may be varied to some extent. Too much soda should be avoided, as any excess tends to make the grease hard. A good proportion is 50 lbs. tallow, 28 lbs. palm oil, 2 lbs. sperm or seal oil, and 12 lbs. soda crystals. Another mixture is 40 lbs. tallow, 40 lbs. palm oil, 4 lbs. whale oil, and 12 lbs. soda crystals, the latter being dissolved in an equal weight of water.

*Rosin Grease.* Take 10 lbs. quicklime, slake well with water and sieve free from grit, stir into 30 lbs. "hard" rosin oil, and allow to stand for 12 hours. By using 20 lbs. "hard" and 10 lbs. "soft" rosin oils, a thinner grease will be got.

By heating rosin grease with rather more oil than is given in the above recipe a clear, transparent, jelly-like mass or grease can be got.

Equal parts of "hard" oil and slaked lime give a hard, granular grease, which cannot well be softened down by the addition of more rosin oil. Not less than two parts of "hard" oil to one of slaked lime is required to give a grease of a good consistence. With "medium" oil  $1\frac{1}{2}$  of oil to 1 of slaked lime gives a grease; with "soft" oil only a thin grease can be got. If larger proportions of oil than those mentioned be used, thin pasty greases are obtained. Much, however, depends on the quality of the oil which is used.

*Tram Grease.* A fine grease is made from 10 lbs. "hard" rosin, 10 lbs. 885 mineral oil, and 10 lbs. slaked lime.

*Axle Grease.* Melt together 14 lbs. palm oil, 22 lbs. anthracene oil, 10 lbs. rosin oil, and 1 lb. soap, keeping the mixture heated until a clear, transparent mass is obtained, then allow to cool.

*Solidified Oil.* Under this name are sold products derived from petroleum and Scotch shale oils, which may be regarded as greases. To make them take 50 lbs. of 885-90 mineral oil, heat to 180° F., then throw in half a pound of soap cut

into fine chips and dried as much as possible by exposure to the air; the heating is kept on until the soap is entirely dissolved in the oil, when the mixture may be allowed to cool down.

*Hot-neck Grease.* A common hot-neck grease can be made from 5 lbs. wool pitch, 20 lbs. brown grease, 30 lbs. hard rosin oil, 40 lbs. dark cylinder oil, and 5 lbs. dry slaked lime, heated together until a homogeneous mass is obtained.

*Colliery Grease.* 50 lbs. rosin oil, 40 lbs. grease oil, 30 lbs. dark cylinder oil, 5 lbs. Yorkshire grease are mixed with 20 lbs. slaked lime.

*Mica Grease.* 50 lbs. rosin oil, 50 lbs. 890-95 Scotch shale oil, 20 lbs. French chalk, and 20 lbs. slaked lime are stirred together.

*Plumbago Lubricant.* 20 lbs. slaked lime, 70 lbs. "hard" rosin oil, 70 lbs. anthracene oil, and 20 lbs. plumbago are stirred together in the usual way.

These are a few of the many recipes which could be given. They will serve to show on what lines to work in making greases.

#### ANALYSIS AND TESTING OF GREASES.

Analysis of greases to ascertain the character and nature of the various constituents is one of a most difficult character, and requires on the part of the analyst a very full knowledge of all the substances which may be used in the preparation of greases, and also requires a good knowledge of the methods by which greases are made. It is not possible to draw up a general scheme by following which any kind of grease may be tested; that given underneath may be taken as a type of a general mode of working, but the analyst must be prepared to devise his own method, according to the character of the results he is obtaining.

Greases vary in character from simple to most complex. They may contain :—

Water.

Soap.

Free alkalies.

Inert mineral matter.

Fatty oil.

Mineral oil.

Pitch.

Coal tar oil.

Rosin oil, etc.

**WATER.** The amount of water in a sample of grease may be estimated by taking 5 grammes in a porcelain crucible, and putting it in an air oven at about 220° F. until it ceases to lose weight. The loss of weight may be taken as the amount of moisture in the grease.

**MINERAL MATTER.** The residue left in the crucible after the determination of the moisture may be heated in the Bunsen burner until all combustible matter has been burnt off. The residue is the mineral matter present in the grease, and this now may be inquired into. The substances most likely to be present are sodium compounds and lime, calcium sulphate, barium sulphate, mica, and French chalk. The residue should be treated with dilute hydrochloric acid, which will dissolve out the sodium compounds and the lime. The other constituents just named will remain behind, being insoluble in the acid, and they can be filtered off. To the acid solution may be added first a little ammonia chloride and then ammonia (if a faint precipitate of alumina be obtained it may be neglected). To the solution is then added some ammonium oxalate; if a white precipitate forms it indicates the presence of lime in the grease. This precipitate may be filtered off and the solution boiled down, when it will give a bright yellow colour to the Bunsen flame



if there be any sodium compounds present. Any inert mineral matter which may be present in the grease will remain in the portion insoluble in the acid, and this is best determined by an examination of its characteristics. If it be French chalk then it will have a soft smooth feel, if it be mica the particles will be more or less flaky and silvery, if it be calcium sulphate it will give a reddish colour to the Bunsen flame, while if it be barytes then the Bunsen flame will be coloured light green.

#### FREE OIL.

Greases contain a large quantity of oil or oily matter of various kinds in the free condition. This is best determined by extraction in a Soxhlett fat extraction apparatus. This apparatus consists of three parts, first a flask, second a Soxhlett fat tube which is shown in Figure 64, and third a vertical Liebig's condenser, these three pieces being connected together in the above order as shown in Figure 65. The grease to be treated is weighed, 10 grammes in a piece of filter paper. It is then placed in the Soxhlett tube, a quantity of petroleum ether is poured into the flask, which is then connected with the Soxhlett tube and this again with the condenser, the flask being placed in a water bath. On heating the latter the ether is caused to vaporise and pass into the condenser, whence it flows back into the Soxhlett tube, where it accumulates and exerts its solvent power upon the free oil in the grease. When the quantity of ether has accumulated to such an extent as to rise above the level

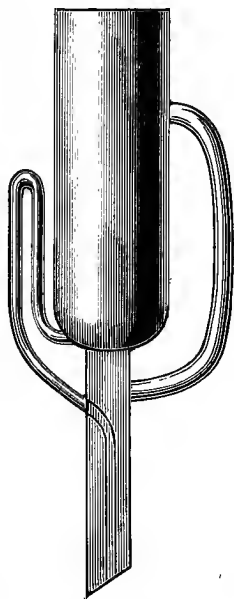


FIG. 64. Soxhlett Fat Extraction Tube.

of the syphon tube, it begins to run off to the flask below with the oil which it has dissolved. The ether is volatilised again and accumulates again in the Soxhlett tube, extracting a further quantity of grease, after which it is ready to pass through the same cycle. Generally it takes about one

to one and a half hours to fully extract the free oil from a sample of grease. The ethereal solution is run into a weighed glass, the ether evaporated, first in a water bath, finally in an air bath, and the residual grease weighed, when it is ready for further examination. It should be examined for free acid, which would tend to indicate the use of Yorkshire grease. For saponifiable fat and for any unsaponifiable fat or oil which may be present, the methods for such tests are described in the chapter on Oil Testing.

The residue which is left in the filter paper in the Soxhlett tube will contain any soap which is present in the grease, together with all mineral matter, which should be examined and the quantity of soap determined by decomposing it with

dilute hydrochloric acid, separating the fatty matter, from the amount of which the corresponding amount of soap may be deducted. The character of the fatty matter can be obtained from an examination of it.

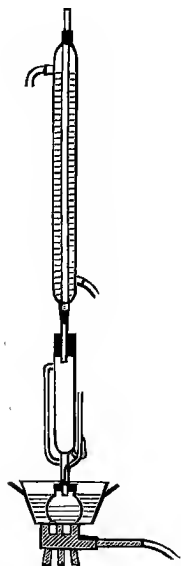


FIG. 65. Fat Extraction Apparatus.

## CHAPTER VIII.

### LUBRICATION.

WHEN two surfaces are caused to rub one upon the other, friction is generated to a greater or less extent. This friction, when it occurs in a machine, is bad, because it leads to excessive wear of the working parts of the machine, and if allowed to go on is likely to lead to other evils, distortion of the machinery, breaking down, etc. It is the object of all machine users to keep this friction down to the lowest possible point. It is not possible to reduce it altogether, but it can and should be kept down to a minimum amount. To reduce this friction, what are called lubricants are applied between the rubbing surfaces, and of all bodies which are capable of being used for this purpose, the oils described in previous chapters give the most satisfactory results.

*Lubricants* should, according to most modern writers on the subject, possess the following properties:—

1. Enough *viscosity* or *body* to keep the surfaces from coming into contact under the maximum pressure which may be applied to them.

Theoretically, if friction results from the rubbing of two surfaces together, and if it is wanted to reduce this, then stress ought to be taken to keep the two surfaces apart.

It is obvious that this is practically impossible, but if we can introduce between them a body, like an oil, which has sufficient adhesive power to adhere to the surfaces, and but little cohesive power in itself, then there would be two films

of lubricant between the surfaces, and the friction which occurs will take place between these two films. Viscosity or body, therefore, is a property of oils which is really dependent on two factors—the cohesion of its particles together, which prevents them being forced asunder by pressure; the adhesion which it has for other bodies, which enables it to adhere to the rubbing surfaces under any pressure.

Bodies may possess great cohesive force but very little adhesive tendency—mercury, for example. These are inadmissible as lubricants. There are bodies which have opposite properties—water may be taken as an example—and although they are more useful as lubricants than the first-mentioned, yet are not wholly satisfactory as lubricants.

2. To have as much *fluidity* as is consistent with the foregoing requirement.

*Fluidity* is an essential condition for a perfect lubricant, for it is only fluids that possess at once that adhesive property above spoken of with the facility of motion of their own particles that will prevent friction. Although solids may have adhesive properties, yet it takes a considerable amount of power to destroy the cohesive force which binds their particles together.

3. A great capacity for storing and carrying away heat. In other words, the property of keeping a bearing cool.

4. A high temperature of decomposition or of evaporation and a low solidification temperature.

These are important essentials. It is obvious that a body cannot be used as a lubricant which would be liable to decompose at, say, 100° C., and probably into constituents which might injure the machine. Again, when a body becomes solid, it loses its lubricating properties in a great degree. Hence, although coconut oil is a good lubricant at high temperatures, yet it cannot be used at ordinary temperatures because of its becoming solid.

5. Freedom from tendency to decompose or oxidise by exposure to air ; or, in other words, free from the liability to gum or clog the bearings.

6. Freedom from acidity and from any tendency to corrode the metal of the bearings to which the oil is applied.

These last two conditions will be dealt with presently. The hydrocarbon oils are the only kind which satisfies the above conditions in the greatest degree.

*Lubrication of machinery* is divisible into two sections :—

A. Lubrication under ordinary atmospheric conditions, which includes the lubrication of all machines, shafting, steam engines, etc.

B. Lubrication at high temperatures and in contact *with steam*, which includes the lubrication of steam and gas engine cylinders and valves.

In dealing with these subjects it will be necessary to repeat in another form what has already been said in previous chapters. This will be done for the sake both of clearness and giving a more complete review of the subject of lubrication.

#### A. LUBRICATION UNDER ORDINARY ATMOSPHERIC CONDITIONS.

The action of the atmosphere on oils is the first point to be considered in connection with their use as lubricants. To some extent what this action is will have been inferred from what has already been said about the properties of oils in previous chapters, but it will be well to repeat in a more definite form the salient points of this important subject.

The atmospheric agencies which act destructively on oils are two, *viz.*, oxygen and water. Some other influences, such as heat, have some action on oils in special places, but as these are of exceeding rare occurrence we can leave them out of account.

The oils, being spread over the bearings in an exceedingly thin film, offer a great surface for the action of these destructive agencies.

Oxygen acts upon all the fixed or fatty oils to a greater or less extent. Some, like linseed, hemp seed, and a few other oils, are greatly affected, while others, like sperm oil, olive, lard, are not much altered. The absorption of oxygen by oils leads to the formation of a hard, dry, resinous mass, which sticks to the bearings, and the formation of this mass is the cause of the "gumming," as it is technically called, of oils. On this account those oils which possess this property in only a slight degree are to be preferred as lubricants. Those oils which, like linseed, dry up entirely are absolutely useless as lubricants, although this very property gives them value to the painter; while other oils which, like cotton or whale oils, do not dry up entirely, but become simply thick and viscid, are not admissible for this purpose. Oxygen does not act on the hydrocarbon oils at all, and mixing a fat with a hydrocarbon oil to some extent prevents the gumming due to the former.

The absorption of oxygen by oils leads to the development of heat. This is greatest when the absorption is greatest. When a little linseed oil is poured over a lump of cotton the amount of surface exposed is very great, and consequently the action of the oxygen is great also. The heat that is developed may rise so high that the cotton will become charred, and not infrequently burst into flames.

This spontaneous combustion of oily cotton is a feature of great interest in the use of oils in textile mills. In these there are always large masses of cotton, wool, etc., lying about in heaps or covering the machine as fluff. Should these become covered, accidentally or otherwise, with oil, and other conditions are favourable, then the oxidation of the oil may go on so rapidly and to such an extent that the fibre will

burst into flame, and a fire is the result. Now, it has been found that only the fatty oils have this property of causing spontaneous combustion; the hydrocarbon oils, having no property of absorbing oxygen and combining with it, cannot cause spontaneous combustion; and, further, it has been shown by Gellatly and others that mixing hydrocarbon with fat oils prevents the latter giving rise to the spontaneous combustion of fibrous bodies.

Thus Gellatly obtained the following results by saturating a handful of cotton waste with various oils, wringing well to get rid of superfluous oil, then placing the oily waste in a chamber kept at a temperature of about 170° F., at which, as we have shown, oxidation begins.

Boiled linseed oil. One sample fired in 75 minutes, another in 105 minutes.

Raw linseed oil. Two samples fired in 4 and 5 hours respectively.

Rape oil. Sample put up at night was found to have been wholly consumed, box and waste, by next morning.

Olive oil. Two samples fired in 5 and 6 hours respectively.

Lard oil fired in 4 hours.

Seal oil fired in 100 minutes.

Sperm oil refused to ignite or to char the waste.

Mineral oils absolutely refused to ignite, and mixtures of 80 rape and 20 mineral, and of 50 seal and 50 mineral, did not, when placed in a warm chamber, develop a temperature sufficient to char the cotton.

It would be well therefore for textile manufacturers to use either pure hydrocarbon oils or mixed oils, and for insurance companies to see that no other oils are used. Then no risks would be run of fires from the spontaneous combustion of oily fibrous materials; for there is no doubt that many textile mill fires have had their origin in this manner.

Moisture acts somewhat differently on the fatty oils—it leads to the splitting up of the oils into their two constituent parts, the base glycerine and the peculiar fatty acids of the oil.

The former is a neutral body and has no corroding action on the metal, neither has it any lubricating properties. The fatty acids have a strong corrosive action on the metal of the machinery. They chemically combine with it, forming a kind of greasy soap which settles in cakes on the machinery and which leads to a rapid and mostly an unequal wear and tear of the machinery. These fatty acids have a much stronger action on brass and copper than on iron.

This decomposing action of moisture is much aided by heat. Hence, when fatty oils are used for lubricating machinery in warm and moist places, there is a great tendency for the machinery to “gum” and for the bearings to be corroded. It would be advisable not to use a fatty oil in such places, or to use one which originally contains any or much free acid.

Hydrocarbon or mineral oils, being perfectly neutral bodies, are not decomposed by water in any way; indeed they undergo no change whatever when exposed to the atmospheric influences to which they are subjected when used for lubricating machinery.

Friction in a machine is caused by the rubbing of its working parts over one another. It is greatest in a badly constructed, roughly put together machine, and is least in well finished machines where the journals, bearings, and other rubbing surfaces have been well polished, and it is greater in a new than an old machine; it is generally in such machines the sole loss of power, and so it is desirable to keep it down as low as possible. In a machine well lubricated there is friction due to the solid working parts of the machine and fluid friction due to the lubricant used.



The friction of solids is of two kinds, *sliding friction* and *rolling friction*. Sliding friction occurs when one flat surface slides over another flat surface; its amount is dependent upon the character of the surfaces, being least with smooth, greatest with rough surfaces; it increases or decreases with an increase or decrease in the pressure. The material of the rubbing surfaces also has some influence, the friction on soft wood is greater than on hardened steel. Rolling friction occurs when one revolving surface rolls on another surface either flat or curved; its amount is dependent on the pressure, speed, and condition of the rolling surfaces.

*Fluid friction* is quite different in its origin, and is therefore subject to different laws than what pertain to the friction of solids. The friction of fluids is quite distinct from the friction of solids; it occurs when a mass of fluid flows through another mass of fluid, and its amount depends very greatly upon the relation in the masses of the fluids and their position. When a fluid flows through a pipe it is found that the fluid in immediate contact with the sides of the pipe flows very slowly, its motion being retarded by contact with the pipe. At the centre the motion is quickest, and it gradually decreases towards the sides, where it is slowest; the same thing happens when a mass of fluid flows through another mass of fluid, which forms the bounding surface at which the friction which occurs causes the speed of flow to be less than in the centre or point farthest removed from the surfaces of contact of the two fluids.

The friction of fluids is independent of the pressure to which they may be subjected, is proportioned to the area of the surface of friction, varies with the velocity, but is independent of the nature of the surfaces with which it is in contact, although the degree of roughness of those surfaces has some influence on the amount of friction.

It varies with different fluids according to their density and relative viscosity.

*The friction of a lubricated surface* is a compound one, part being due to the lubricant and part due to the rubbing surfaces. When well lubricated it obeys the laws of fluid friction to a great extent. On the other hand, a badly lubricated surface is governed by the laws of solid friction. With very heavy pressures and slow speeds, the moving part and its bearing are forced into close contact, and there is a considerable wear of the surfaces, while in the case of light pressures and high velocities, such as are met with in the spindles of a spinning frame, the moving surface floats on a film of the lubricant, and the friction thus occurs between two layers of fluid, one in contact with each surface. These cases represent the extreme limits met with in the lubrication of machinery, one limit being that of purely solid friction where great friction and resistance are met with, the other where the friction and resistance are entirely due to the fluid lubricant, and which generally separates completely the surfaces of the solids.

The factors which govern the selection of a lubricant for particular purposes are *speed, pressure, temperature*.

1. *Speed*. This is a most important factor in guiding consumers in selecting oils for lubricating a machine. The rule is a very simple one, the quicker the speed the thinner and lighter is the oil that is required. If an oil with a great viscosity were used for lubricating spindles, for instance, which revolve at from 6000 to 8000 revolutions per minute, the cohesive force which holds the particles of oil together is too great and cannot be overcome with sufficient rapidity at the speed of revolution, and so a certain amount of friction is introduced which tends to reduce the speed of the spindle, which is not desirable. On the other hand, with a slow motion an oil that would give good results when used on a

spindle would fail, because it would not have sufficient viscosity to remain on the bearings.

2. *Pressure.* This is equally as important a factor as speed. Where the pressure is light, as in the foot-steps of spindles, bearings of small levers, a light oil of low viscosity is required, and will be found sufficient to keep the surfaces apart and prevent friction. With heavy pressures, as on engine shaft bearings, the bearings of calender bowls, etc., a heavy, high viscosity oil is required; one which has great cohesive and adhesive properties is especially required, so that it cannot be pressed out from between the bearings under the heaviest pressure to which it is subjected.

*Speed* and *pressure* are intimately connected. High speeds are nearly always associated with low pressures, and *vice versa*. High pressures are nearly always combined with slow speeds, although there is much variety of conditions of speed and pressure under which a machine can be worked.

3. *Temperature.* This is also an important factor to be considered in selecting oils. An oil that would work satisfactorily in a cold place would not give equally satisfactory results in a hot room. The heat would cause the oil to lose some of its viscosity and to flow too freely from the bearings, and hence not lubricate them properly. In hot places a more viscous oil can be used than should be selected for cold places. Again, an oil that is used in such places as the turning shops of iron works, and for lubricating outside bearings and motions of all kinds, should have a low setting point or cold test. For such purposes there are no better oils than the Russian petroleum oils, which have low setting points, while of the fatty oils, sperm, rape, and castor are the best for cold places.

In selecting oils for the lubrication of machinery the following hints will serve to guide consumers and dealers in their selection of a suitable oil:—

Under very great pressures and slow speeds: Blacklead, talc mixed with tallow if in a warm place, or with castor oil or Russian petroleum oil in a cold place.

Under heavy pressures and slow speeds, such as the bearings of engine shafts, calender bowls, crushing mills: Lard, tallow if used in warm places, or castor oil, heavy American petroleum oils of 910-920 gravity, and Russian petroleum oils in cold places.

Heavy pressures and high speeds: Rape, olive, and lard oils or mixtures of these with heavy American oils of 905-910 gravity for warm places; rape, 905-910 American and light Russian petroleum oils in cold places.

Light pressures and high speeds, such as the spindles of textile spinning frames, etc.: Sperm, olive, rape, heavy Scotch shale oils of 885-890 gravity, or mixtures of light shale and petroleum oils of 880-900 gravity with sperm, rape, olive, coconut, lard, or other fat oils.

For shafting and all other ordinary machinery bearings: Olive, rape, lard, heavy shale, medium American petroleum and the thinner Russian oils, either alone or mixed together, can be used with very satisfactory results. In making the selection regard should be paid to the average working temperature of the place or works. A pure hydrocarbon oil or a mixed hydrocarbon and fat oil will give far better results than can be obtained by using a pure fat oil.

It should be *the rule* to use the thinnest oil that will reduce the friction of the machinery to the minimum amount, as the use of too heavy an oil simply means the use of extra power, which is not economical. In those cases, as in the bearings of marine engine shafts, slides and screw shafts, where, owing to incessant use day after day without stopping, there is considerable tendency to heating, it will be found best to use a pure mineral oil. Give a very liberal feed, collect the surplus oil in suitable drip tins, allow this to stand,

or filter it from any dirt or grit, and use it again. The question arises whether it would not be worth while for marine engineers to invent a form of hollow bearings through which a current of cold oil or water could be passed. The former would be preferable.

*A machine* will contain within itself both quick and slow speeds, and heavy and light pressures; now it will follow, attending to the rules laid down, that to efficiently lubricate such a machine, a spinning frame for instance, a variety of oils would be needed, a thin oil for the quickest bearings, a thick oil for the heaviest and slowest bearings and medium oils for intermediate pressures and speeds; practically it will be found impossible to carry out this provision of oils to its fullest extent, as workmen cannot always be trusted to use the right kind of oil in the right place, but at least two kinds of oil should be provided—a light oil for the quicker motions and a heavier oil for the slower motions.

No solid oil or grease should be used for lubricating ordinary machinery for reasons already given. It is on record that a mill in America which used to be lubricated entirely by greases was with difficulty driven by the water-wheel; replacing the grease with a mineral lubricating oil it was found that there was quite sufficient power to drive the machinery, and experiments showed that it took 25 per cent. less power to drive the machinery with oil than with grease and the general temperature of the machinery was 35 less. Oil dealers are in the habit of talking to their customers a good deal of twaddle about oils, much of which arises from their ignorance of the real properties of oils and of lubrication; they will enlarge on the merits of having an oil of high flash point, arguing that it must be safer than an oil of low flash point. Well, so it is, but flash point is not everything in oils; it may be accepted that for ordinary machinery bearings any oil flashing above 330° F., and

for spindles any oil flashing above 350° F., is safe enough. Now, high flash point is invariably associated with high viscosity, and if for fast and light machinery, such as spindles, it is necessary to keep the viscosity low, then the flash point must be low too; in fact both oil dealers and consumers should make viscosity, not flash point, the real crux or test point of oils; the great objection to this so far has been the want of a standard apparatus for the determination of viscosities. This, however, has now been remedied.

Some dealers have much to say about the body of their oils. They are continually aiming at having great body, and will offer their customers for the lubrication of spindles an oil having a body or viscosity high enough for an engine shaft. This is pushing the matter too far to the other extreme. If viscosity or body is to be dilated upon, it should be done in an intelligent manner, and the viscosity of an oil should be carefully adapted to the particular purpose for which the oil is to be used.

Marine engineers have a fondness for the oils they use on the slides and other bearings to work what they call soapy or with a froth, being under the opinion that the bearings are being efficiently lubricated when this occurs. This is a mistaken notion for two reasons, because efficient lubrication can be got without soaping or frothing, and because soaping and frothing cannot be got without the use of rather poor qualities of fat oils.

Oils are also sold under a variety of fancy names. This is all very well, and there is no objection to a dealer giving any name he likes to his oils. The great objection is charging a fancy price for such oils, far beyond their actual worth.

The production of stains in cloths by the oils used for the lubrication of textile machinery of various kinds is one of great importance, and the necessity of avoiding the production of such stains has led to the introduction by dealers of

“stainless oils” for the lubrication of spinning frames and weaving machinery. Some of these are correctly named, others are mere rubbish and are “stainless” only in name. Oil stains on cloth arise in several ways. However much care be taken in the working of the textile machinery, it is always impossible to prevent accidental splashes of the oil on the yarn or cloth. The modern spindle practically revolves in a bath of oil. Some of this, by the centrifugal motion, cannot fail, with many forms of spindle, to be thrown on the yarn as it is being spun. To prevent this to a great extent foot-step protectors have been invented. Those made by Messrs. E. Jagger & Co. of Oldham are very efficient in preventing splashing of oil. Then in the loom there is a liability of oil dropping from overhead motions on to the warp or woven cloths. These oil stains are most undesirable, as they spoil the appearance of the cloth. They must be got out by some means. Now it has been found that these oil stains can be divided into two kinds, one that can be easily got out by treatment with alkaline liquors, such as boiling with solutions of soda ash, caustic soda, and lime, and another kind of stain that cannot be got out by these, practically the only cleansing processes that can be applied to textile fabrics. These fixed stains are of two kinds, one oily, evidently due to the oil used, the other of a metallic—chiefly iron—origin. The latter stains have probably got on to the fabrics in this way. The friction of the machinery causes the wearing down and the production of a fine metallic dust from the metal of the bearings. This gets into the oil, and, of course, if any of this gets on the fabrics the process of cleansing does not affect them, in fact it tends to fix them on the cloth, while the oil is washed away. They can only be removed by treatment with acids. As they are derived from the metal of the bearings, oils that are acid, and hence liable to corrode the metal, are more liable

to produce them than pure neutral oils. Iron stains are red, and can be tested for in this way. Treat the stain with a drop of hydrochloric acid, and then a drop of a solution of ferrocyanide (yellow prussiate) of potassium, when a blue colour, showing the presence of iron, will be obtained. Copper or brass stains may be of various colours—black, grey, or greenish. Treated as with iron stains, they will give a brown colour, or, if first with hydrochloric acid, then with a drop of ammonia, they will give a blue colour, turning black on adding a drop of ammonia sulphide.

*Mineral oils* are the only oils that give a permanent stain on fabrics. If a drop of such oil gets on to textile fabrics, whether it be a so-called "bloomless" or "stainless" oil or not, it cannot be got rid of entirely. How much is got rid of depends upon the length of time which elapses between the oiling of the goods and the cleansing operation. The longer the interval, the more the oil has the chance of penetrating into the fibres of the cloths or yarns, and the more it resists the cleansing process. The only way of removing such stains is to cover them with olive oil, allow this to soak well in, and then scour with soda lye in the usual way. Even then it is sometimes difficult to remove them entirely. Stains caused by mixed oils are removed more easily, but it has been found by Scheurer that the proportion of mineral oil must not exceed 50 per cent. of the mixed oil. Even then it requires prolonged treatment to remove all the oil. From which it will be seen that no mineral oil, or even a mixed oil, can really be considered, nor should they be sold, as "stainless oils," although such is done too often. Some qualities of these "stainless oils" are very pale in colour, almost white. They impart no or but slightly visible stains, and hence in a sense justify their name. A lubricating oil containing more than 25 per cent. of mineral oil cannot be considered as a "stainless oil,"



because it is impossible to ensure the entire removal of the oil stains it may produce if it contains more than that amount.

Cotton cloths have to be bleached, and they pass through a very severe treatment with alkali and chlorine during the process. If any mineral oil is left in the cloths after the treatment with alkali, the chlorine attacks it, and forms decomposition products.

These are at first white, after some time become yellow, and thus spoil the appearance of the fabrics. Such stains are difficult to remove, and the only practicable method is to saturate them with olive oil, and, after soaking for some time, boiling with alkaline lyes.

It will be found best to take proper precautions to prevent splashing of the oils from bearings on to the cloths, etc., while they are being spun or woven, and there is more chance of success by so doing than by using nine out of ten samples of stainless oils.

#### B. LUBRICATION AT HIGH TEMPERATURES.

The second part of lubrication deals with the lubrication of steam and gas engine cylinders, a subject of great importance to users of engine power who, and especially their engineers, have very little knowledge of the scientific principles that underlie the lubrication of engine cylinders, and very few oil dealers are in a position to properly advise their clients on this matter.

The conditions to which oils are subjected when used for the lubrication of a cylinder are those to which they might be subjected under ordinary circumstances, and which have already been dealt with, and three new conditions, *viz.*: heat, pressure, and moisture; and these are the main factors to be considered.

Oils which are not altered under ordinary conditions, and

are therefore efficient lubricants for machinery, are much affected by the increased heat and pressure to which they are subjected, and to a greatly increased action of water when used in steam engine cylinders.

The temperature to which the oils are subjected varies with the pressure, and the following table shows the relation of temperature to pressure :—

Pressure above Zero.	Approximate Pressures above Atmosphere.	Temperature of Steam and Water.	Thermal Units contained in One Pound.	
			Contained in the Water.	Latent Heat of Steam.
Lbs.	Lbs.	Deg.		
20	5.5	228.0	280.1	952.8
25	10.5	240.1	241.3	945.3
30	15.5	250.4	251.9	937.9
35	20.5	259.3	260.9	931.6
40	25.5	267.3	268.9	926.0
45	30.5	274.4	276.2	920.9
50	35.5	281.0	282.8	916.3
55	40.5	287.1	289.0	912.0
60	45.5	292.7	294.7	908.0
65	50.5	298.0	300.1	904.2
70	55.5	302.9	305.0	900.8
75	60.5	307.5	309.7	897.5
80	65.5	312.0	314.2	894.3
85	70.5	316.1	318.5	891.4
90	75.5	320.2	322.6	888.5
95	80.5	324.1	326.5	885.8
100	85.5	327.9	330.3	883.1
105	90.5	331.3	333.7	880.7
110	95.5	334.6	337.2	878.3
115	100.5	338.0	340.6	875.9
125	110.5	344.2	346.9	871.5
135	120.5	350.1	352.8	867.4
145	130.5	355.6	358.4	863.5
155	140.5	361.0	363.8	859.7
165	150.5	366.0	368.7	856.2
175	160.5	370.8	373.5	852.9
185	170.5	375.3	378.2	849.6
195	180.5	379.7	382.7	846.5
210	195.5	386.0	389.2	841.9

The effect of temperature on oils is first to cause them to become thin and watery, or, as the oil dealers say, attenuate; this means that they lose much of their lubricating power,

and hence it follows that those oils which suffer the least loss of viscosity under the influence of heat must be best. Oils vary much in this respect. Some, like sperm, thin down very little; mineral oils attenuate very much—the Russian oils being the worst sinners in this respect. This loss of viscosity by heat decreases the adhesiveness of the oils for the hot surfaces, hence there is more tendency for hot surfaces to work “dry” than for cold surfaces. Experience has shown that fat oils lose very much of their adhesive power by heat, distilled mineral oils next, and that a steam-refined and filtered natural cylinder oil loses the least in proportion. This is therefore a more efficient cylinder lubricant than either of the other kinds.

TABLE OF VISCOSITIES OF OILS.

	Temperature—Fahrenheit.				
	70°	100°	120°	150°	180°
Castor Oil . . . . .	1248	487·5	201·5	91	48
Thickened Rape Oil . . . . .	1370	331·5	279·5	156	78·5
Sperm Oil . . . . .	58·5	36·4	26	19·5	17
Colza Oil . . . . .	131	56	44	32·5	28
Whale Oil . . . . .	128·7	61	44	28·5	28
Tallow Oil . . . . .	105	63	45	30	20
Cotton Oil . . . . .	100	55	40	25	20
American 885 Oil . . . . .	68	35	23	15	14
American 905 Oil . . . . .	113	44	32·5	19·5	18
American 915 Oil . . . . .	140	47	36	21	19·5
Scotch 865 Oil . . . . .	32·5	22	18	15·5	13
Scotch 885 Oil . . . . .	58·5	26	22	18	15·5
Scotch 890 Oil . . . . .	71·5	39	26	19·5	17
Russian 906 Oil . . . . .	292·5	97·5	56	30	22
Russian 911 Oil . . . . .	462	143	91	82·5	26
Rosin Oil, dark . . . . .	152·5	97·5	38	22	18
Rosin Oil, pale . . . . .	136·5	49·4	25	18	17
Cylinder Oil, medium . . . . .	...	385	255	170	70
Cylinder Oil, pale . . . . .	...	405	265	120	90
Cylinder Oil, dark . . . . .	...	890	495	230	100

Before the cylinder oils made from petroleum oils were introduced, the most commonly used oils and fats for this

purpose were tallows, suet, neatsfoot and castor oils. These were the favourites with engineers, although occasionally other oils were used. These and all fat oils should *never* be used for the purpose.

The reason for this can be sought in the chemical compositions of the oil or fat. These are, as already explained, *glycerides*; and when they are brought under the conditions present in a steam engine cylinder, heat, and pressure in the presence of water, they are split up into their two constituent parts—the base *glycerine* and the fatty acid peculiar to the particular fat or oil used. The glycerine being volatile in the presence of steam passes away with the exhaust steam; the fatty acid not being volatile remains behind in the cylinders and settling on the metal begins to corrode it, forming solid cakes and balls of a black greasy substance consisting of fat and grease, fatty acid, partly free and partly in combination with iron, particles of metallic iron. It is no uncommon thing to take large masses of this stuff out of a cylinder.

This corrosion leads to pitting of the working and other surfaces of the cylinder; the friction and therefore the wear of the rubbing surfaces are increased, which means an increase in the cost of repairs. It is no uncommon thing to take out bolts which have been corroded away to a mere thread by the action of the fat acids which have been formed by the decomposition of the fats and oils used. This evil is intensified where suet is used, for here the animal tissue which is present in suet is an additional source of trouble, as it gets into the interior of the cylinder and clogs the piston in its working, besides having a greater tendency to cause the formation of cylinder grease balls.

It is evident therefore that fat oils, such as tallow, lard, castor oil, neatsfoot oil, are not suitable for use as lubricants for steam engine cylinders, and their use should be abandoned.

When after using such oils a change is made to a mineral cylinder oil, the cakes of grease, etc., begin to soften and gradually work out, and the cylinder gets foul very quickly. Hence users are often led to think that mineral oils work dirty and give up their use ; this is a mistake, the use of the oil should be persevered in, the cylinder will gradually become freed from the residues of fatty matter and then these oils work as clean as an oil can be expected to work. In gas engine cylinders the evils attending the use of fat oils which are found in steam engine cylinders are much intensified by the oil being subjected to greater heat and to risk of burning from the flame of the explosion, whereby it is subjected to a burning action leading to the partial combustion of the oil, evolving unpleasant odours and the deposition of solid carbon on the working surface. This undesirable feature of the decomposition of oils is in addition to being split up into their base, glycerine, and the fatty acid, so that if fatty oils are objectionable to use for steam engine cylinders they are still more objectionable for use in gas engine cylinders. In the hydrocarbon cylinder oils, fortunately, the engineer has a variety of oils that are free from the objections attending the use of fatty oils for the lubrication of engine cylinders. These oils are obtained from petroleum.

Of these petroleum cylinder oils there are two kinds : 1st, dark cylinder oils of great body obtained from the natural oils by a process of distillation to free them from the more volatile oils, by refrigeration and filtering to free them from paraffin and grit. They vary in consistency : some are viscid fluids, others are of a buttery consistency, some are of a dark colour varying from brown to black, others are pale brownish yellow, and all have a strong greenish fluorescence or bloom ; these variations are dependent partly on the quality of the natural oil from which they are obtained

and partly on the process of refining to which they have been subjected.

They differ very much in their lubricating properties. Some makes are very solid at low temperatures, but when heated become very thin and have little lubricating property; and this wax, although solid at ordinary temperatures, becomes very thin at high temperatures and it has no lubricating properties whatever; hence all cylinder oils should be free from this body.

These oils have a low specific gravity, 0·896 to 0·905; flash at from 500° to 540° F.

2nd, pale cylinder oils. These are prepared by distillation from the residuum; they are always of a brownish yellow colour, fluid. Although used for the purpose, yet they have not sufficient body to make them efficient lubricants for cylinders. These pale oils usually range about ·925 to ·930 in specific gravity and flash at about 430° F. Cylinder oils are also sold under various fancy names, such as vaseline, vaseline tallow, valvoleum, etc. These are generally mixed oils either of different qualities of hydrocarbon oils or of hydrocarbon with fat oils; some are good, others bad, many rather indifferent lubricants.

Cylinder oils ought to have the following properties: A high flash point. This should never be less than 475° F., although some authorities advocate 500° F., but the writer is no advocate of unreasonably high flash points, as he thinks they can only be obtained at the expense of losing some other valuable property of the oil. Cylinder oils ought to lose little or nothing when kept at a temperature of 212° F. for twenty-four hours. An oil which loses under such circumstances more than say 0·5 per cent. ought to be rejected. Their viscosity should be as high as possible, consistent with ability to flow, while they should lose as little as possible on heating to 212° F. They should have the property of

adhesiveness to hot metal surfaces, so that they will flow freely over all the interior working surfaces of the cylinder and valve chamber and lubricate them properly. There is a great difference in oil in this respect. The natural cylinder oils obtained by treatment with superheated steam and filtration are superior to the distilled cylinder oils in many respects, chiefly because they do not lose their viscosity so readily on heating. Cylinder oils made by mixing fat oils and hydrocarbon oils together should never be used, for admixture with mineral oils does not prevent the decomposition of the fat oil into its baneful constituent, and the dealer who largely trades in such mixed oils generally uses a poor quality of hydrocarbon oil, hoping to conceal its inferiority by using a large proportion of neatsfoot oil or tallow. The writer has had samples of such oils submitted to him for testing which he would have been sorry to use on a shaft, let alone in the cylinder of a steam engine.

*Gas engine cylinders and hot air engine cylinders* can be lubricated much on the same lines and with the same oils as a steam engine cylinder, the conditions of both being nearly identical, a new condition of a hotter and drier temperature being present. Within the gas engine the presence of flame in the cylinder has already been mentioned. This action leads to more or less charring. This is greater with fat oils than with mineral oils owing to the non-volatile character of the former, while the latter volatilise before they char. The oil *par excellence* for lubricating gas engine cylinders is a *pure* hydrocarbon oil having a high vaporising point about 260° F., a flash point of 430° F., and a fire test of 550° F.





## APPENDIX A.

### DENSITIES CORRESPONDING TO BAUME'S HYDROMETER FOR LIQUIDS LIGHTER THAN WATER.

B.	Density.	B.	Density.	B.	Density.	B.	Density.
10	1.000	23	0.918	36	0.849	49	0.789
11	0.993	24	0.913	37	0.844	50	0.785
12	0.986	25	0.907	38	0.839	51	0.781
13	0.980	26	0.901	39	0.834	52	0.777
14	0.973	27	0.896	40	0.830	53	0.773
15	0.867	28	0.890	41	0.825	54	0.768
16	0.960	29	0.885	42	0.820	55	0.764
17	0.954	30	0.880	43	0.816	56	0.760
18	0.948	31	0.874	44	0.811	57	0.757
19	0.942	32	0.869	45	0.807	58	0.753
20	0.936	33	0.864	46	0.802	59	0.745
21	0.930	34	0.859	47	0.798	60	0.749
22	0.924	35	0.854	48	0.794		

## APPENDIX B.

## COMPARISON OF DIFFERENT THERMOMETRIC SCALES.

Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.
-40	-40	2	35.6	44	111.2	86	186.8
39	38.2	3	37.4	45	113	87	188.6
38	36.4	4	39.2	46	114.8	88	190.4
37	34.6	5	41	47	116.6	89	192.2
36	32.8	6	42.8	48	118.4	90	194
35	31	7	44.6	49	120.2	91	195.8
34	29.2	8	46.4	50	122	92	197.6
33	27.4	9	48.2	51	123.8	93	199.4
32	25.6	10	50	52	125.6	94	201.2
31	23.8	11	51.8	53	127.4	95	203
30	22	12	53.6	54	129.2	96	204.8
29	20.2	13	55.4	55	131	97	206.6
28	18.4	14	57.2	56	132.8	98	208.4
27	16.6	15	59	57	134.6	99	210.2
26	14.8	16	60.8	58	136.4	100	212
25	13	17	62.6	59	138.2	101	213.8
24	11.2	18	64.4	60	140	102	215.6
23	9.4	19	66.2	61	141.8	103	217.4
22	7.6	20	68	62	143.6	104	219.2
21	5.8	21	69.8	63	145.4	105	221
20	4	22	71.6	64	147.2	106	222.8
19	2.2	23	73.4	65	149	107	224.6
18	0.4	24	75.2	66	150.8	108	226.4
17	+1.4	25	77	67	152.6	109	228.2
16	3.2	26	78.8	68	154.4	110	230
15	5	27	80.6	69	156.2	111	231.8
14	6.8	28	82.4	70	158	112	233.6
13	8.6	29	84.2	71	159.8	113	235.4
12	10.4	30	86	72	161.6	114	237.2
11	12.2	31	87.8	73	163.4	115	239
10	14	32	89.6	74	165.2	116	240.8
9	15.8	33	91.4	75	167	117	242.6
8	17.6	34	93.2	76	168.8	118	244.4
7	19.4	35	95	77	170.6	119	246.2
6	21.2	36	96.8	78	172.4	120	248
5	23	37	98.6	79	174.2	121	249.8
4	24.8	38	100.4	80	176	122	251.6
3	26.6	39	102.2	81	177.8	123	253.4
2	28.4	40	104	82	179.6	124	255.2
1	30.2	41	105.8	83	181.4	125	257
0	32	42	107.6	84	183.2	126	258.8
+1	33.8	43	109.4	85	185	127	260.6

## COMPARISON OF DIFFERENT THERMOMETRIC SCALES

*(Continued).*

Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.
128	262.4	178	354.2	229	446	280	537.8
129	264.2	179	356	230	447.8	281	539.6
130	266	180	357.8	231	449.6	282	541.4
131	267.8	181	359.6	232	451.4	283	543.2
132	269.6	182	361.4	233	453.2	284	545
132	271.4	183	363.2	234	455	285	546.8
133	273.2	184	365	235	456.8	286	548.6
134	275	185	366.8	236	458.6	287	550.4
135	276.8	186	368.6	237	460.4	288	552.2
136	278.6	187	370.4	238	462.2	289	554
137	280.4	188	372.2	239	464	290	555.8
138	282.2	189	374	240	465.8	291	557.6
139	284	190	375.8	241	467.6	292	559.4
140	285.8	191	377.6	242	469.4	293	561.2
141	287.6	192	379.4	243	471.2	294	563
142	289.4	193	381.2	244	473	295	564.8
143	291.2	194	383	245	474.8	296	566.6
144	293	195	384.8	246	476.6	297	568.4
145	294.8	196	386.6	247	478.4	298	570.2
146	296.6	197	388.4	248	480.2	299	572
147	298.4	198	390.2	249	482	300	573.8
148	300.2	199	392	250	483.8	301	575.6
149	302	200	393.8	251	485.6	302	577.4
150	303.8	201	395.6	252	487.4	303	579.2
151	305.6	202	397.4	253	489.2	304	581
152	307.4	203	399.2	254	491	305	582.8
153	309.2	204	401	255	492.8	306	584.6
154	311	205	402.8	256	494.6	307	586.4
155	312.8	206	404.6	257	496.4	308	588.2
156	314.6	207	406.4	258	498.2	309	590
157	316.4	208	408.2	259	500	310	591.8
158	318.2	209	410	260	501.8	311	593.6
159	320	210	411.8	261	503.6	312	595.4
160	321.8	211	413.6	262	505.4	313	597.2
161	323.6	212	415.4	263	507.2	314	599
162	325.4	213	417.2	264	509	315	600.8
163	327.2	214	419	265	510.8	316	602.6
164	329	215	420.8	266	512.6	317	604.4
165	330.8	216	422.6	267	514.4	318	606.2
166	332.6	217	424.4	268	516.2	319	608
167	334.4	218	426.2	269	518	320	609.8
168	336.2	219	428	270	519.8	321	610.6
169	338	220	429.8	271	521.6	322	612.4
170	339.8	221	431.6	272	523.4	323	614.2
171	341.6	222	433.4	273	525.2	324	616
172	343.4	223	435.2	274	527	325	617.8
173	345.2	224	437	275	528.8	326	619.6
174	347	225	438.8	276	530.6	327	621.4
175	348.8	226	440.6	277	532.4	328	623.2
176	350.6	227	442.4	278	534.2	329	625
177	352.4	228	444.2	279	536	330	626.8

## COMPARISON OF DIFFERENT THERMOMETRIC SCALES

*(Continued).*

Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.
331	628·6	339	643	347	657·4	355	671·8
332	630·4	340	644·8	348	659·2	356	673·6
333	632·2	341	646·6	349	661	357	675·4
334	634	342	648·4	350	662·8	358	677·2
335	635·8	343	650·2	351	664·6	359	679
336	637·6	344	652	352	666·4	360	680·8
337	639·4	345	653·8	353	668·2		
338	641·2	346	655·6	354	670		

## APPENDIX C.

TABLE OF SPECIFIC GRAVITIES OF FATTY OILS AT 15° C. (60° F.).

		Weight of 1 gallon.	Trade Weight per gallon.
Almond Oil . . . . .	0·919	9 lb. 3 oz.	9 lb.
Arachis (Ground Nut) Oil . . . . .	0·920	9 " 3 "	9 $\frac{1}{2}$ "
Castor Oil . . . . .	0·964	9 " 9 $\frac{1}{2}$ "	9 $\frac{1}{2}$ "
Coconut Oil . . . . .	0·925	9 " 4 "	...
Cotton Seed Oil . . . . .	0·923	9 " 4 "	9 $\frac{1}{4}$ "
Linseed Oil . . . . .	0·932	9 " 5 $\frac{1}{2}$ "	9 $\frac{1}{4}$ "
Olive Oil . . . . .	0·915	9 " 2 $\frac{1}{2}$ "	9 "
Palm Oil . . . . .	0·9406	9 " 6 "	...
Rape Oil . . . . .	0·914	9 " 2 "	9 "
Sesame Oil . . . . .	0·923	9 " 4 "	9 $\frac{1}{4}$ "
Lard Oil . . . . .	0·912	9 " 2 "	9 "
Tallow Oil . . . . .	0·912	9 " 2 "	9 "
Neatsfoot Oil . . . . .	0·914	9 " 2 $\frac{1}{2}$ "	9 "
Tallow . . . . .	0·940	9 " 6 "	...
Sperm Oil . . . . .	0·883	8 " 14 "	8 $\frac{3}{4}$ "
Whale Oil . . . . .	0·925	9 " 4 "	9 $\frac{1}{4}$ "
Mineral Oil . . . . .	0·875	8 " 12 "	8 $\frac{3}{4}$ "
Mineral Oil . . . . .	0·903·7	9 " 1 "	9 "
Mineral Oil . . . . .	0·925	9 " 4 "	9 "

In the summaries of the constants given under each oil, gravities at other temperatures are given.

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